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The Journal of the SOCIETY OF DYERS AND COLOURISTS

Volume 71 Number 5

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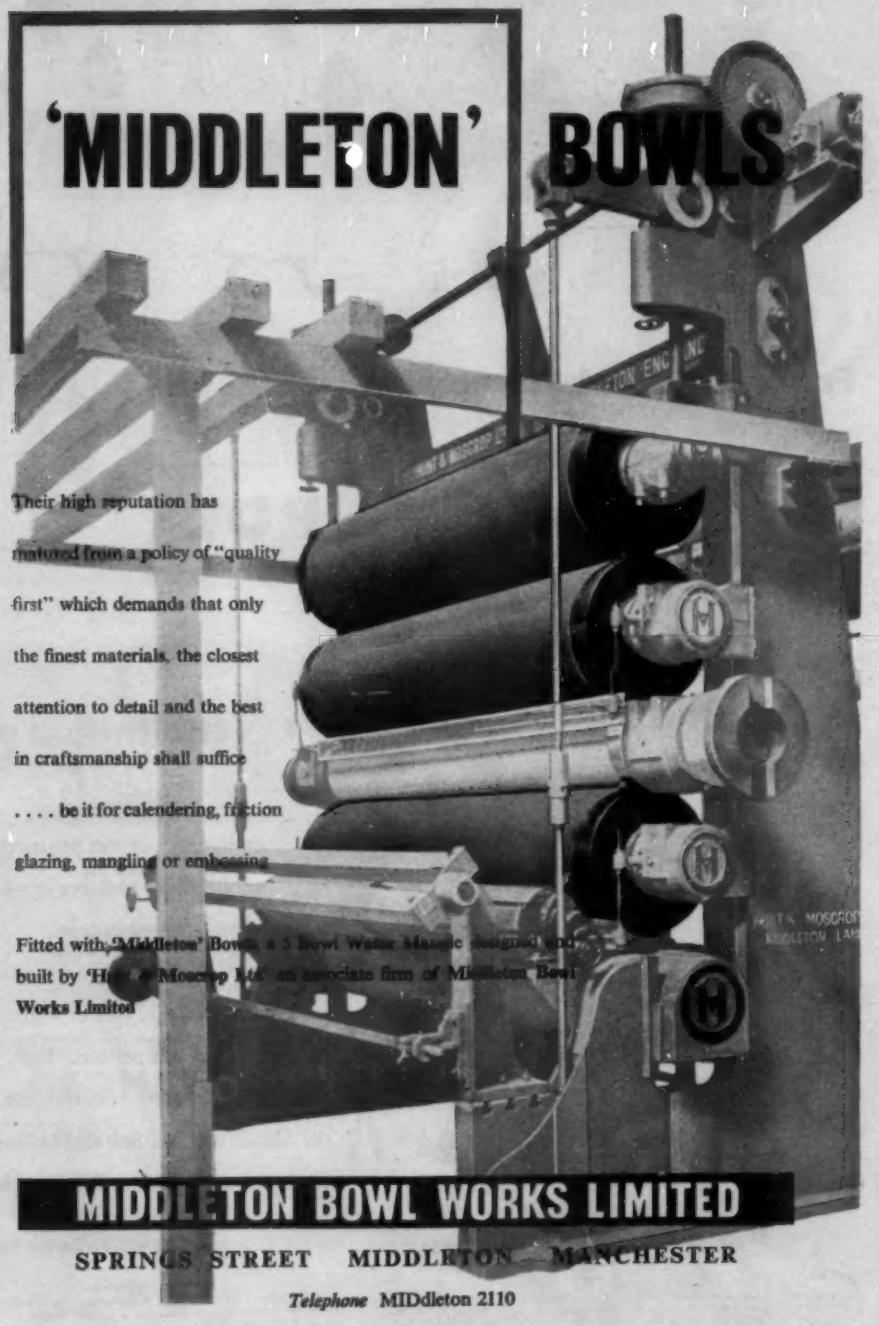

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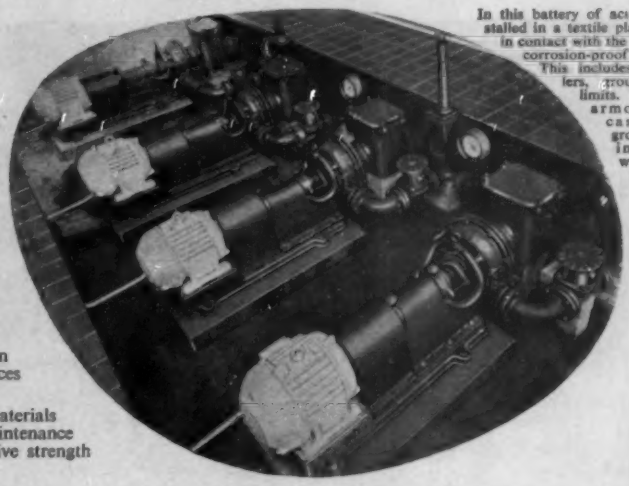

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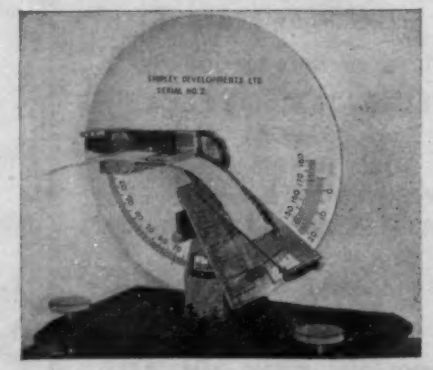

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–6 of the January 1955 and pages 262–266 of the July 1954 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138–9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

The Dyeing of Wool–Terylene Mixture Materials	*A. S. Fern and H. R. Hadfield*
The Dyeing of Terylene Polyester Fibre with Disperse Dyes above 100°C.	*A. S. Fern*
The Dyeing of Textile Fibres at Temperatures above 100°C.	*J. A. Fowler*
Textile Printing in London and the Home Counties	*D. King*

COMMUNICATIONS

The Reflectance of Dyed Fabrics in relation to Dye Concentration	*E. Atherton*
The Polarography of Azo Dyes	*J. de O. Cabral and H. A. Turner*
Some Aspects of the Mechanical Finishing of Cotton Textiles	*G. M. Nabar and G. P. Tawde*

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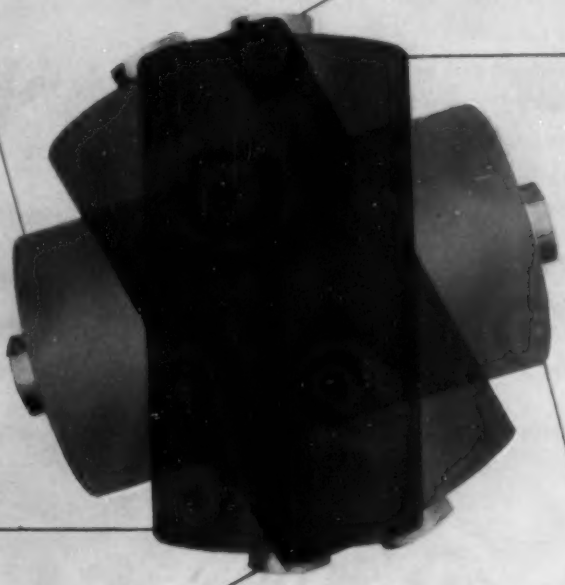

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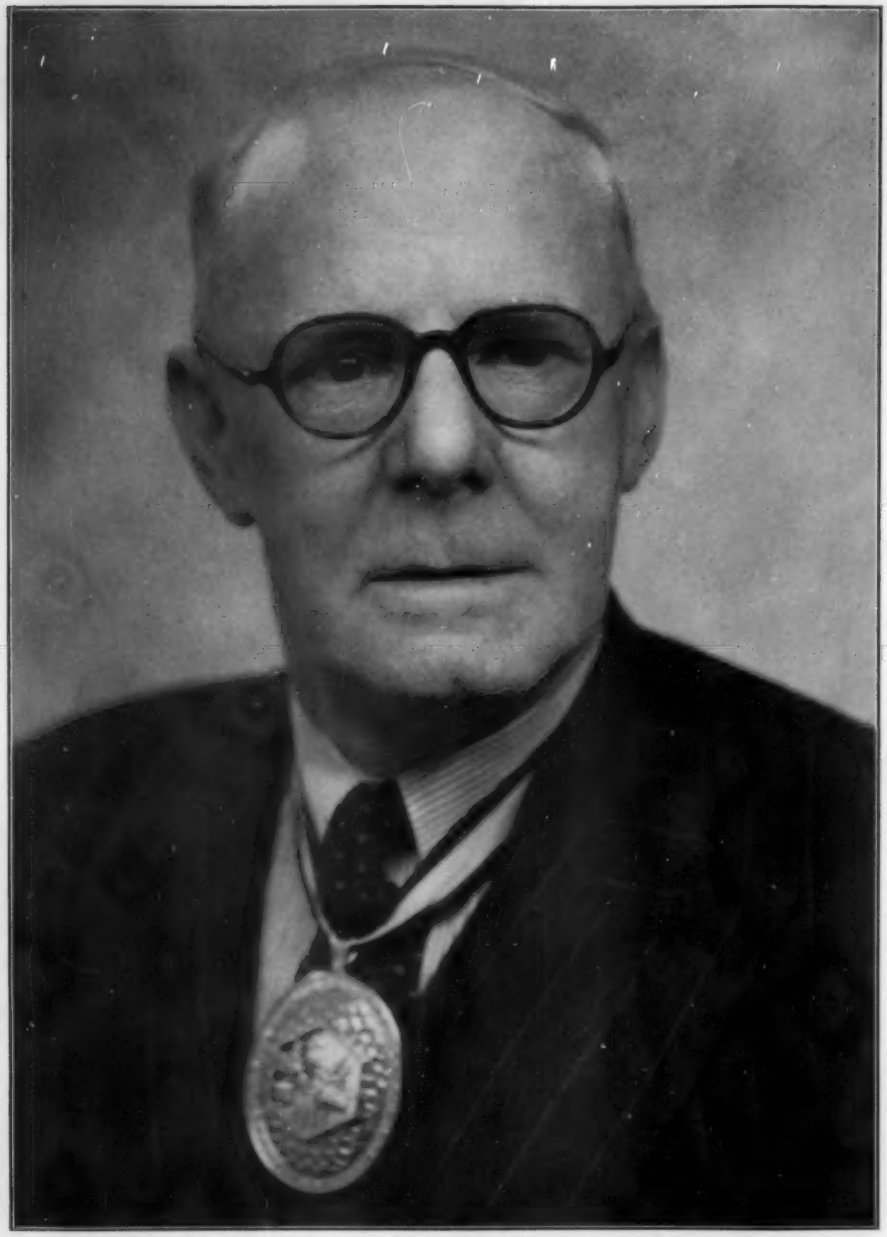


FRED SMITH F.S.D.C.

Re-elected President of the Society of Dyers and Colourists 1955

THE JOURNAL

OF THE

Society of Dyers and Colourists

Volume 71 Number 5 MAY 1955 *Issued Monthly*

Proceedings of the Society

Annual General Meeting

The Seventy-first Annual General Meeting of the Society of Dyers and Colourists was held at the Queens Hotel, Leeds, on Friday, 1st April 1955, at 5.0 p.m., the President (Mr. FRED SMITH) being in the chair and about sixty members of the Society being present.

In opening the meeting, the President paid tribute to the late Mr. F. L. Goodall, who had passed away the previous June, when in office as President.

The minutes of the Seventieth Annual General Meeting, held on 30th April 1954 at the Midland Hotel, Manchester, were read by the General Secretary (Mr. J. W. NICHOLLS), together with the Minutes of the Extraordinary General Meeting held on 8th September 1954 at the Victoria Hotel, Bradford.

Mr. C. O. CLARK proposed, Mr. H. A. TURNER seconded, and it was agreed that the minutes of both meetings be accepted as true records and signed.

REPORT OF THE COUNCIL

The Presidential Message on the occasion of the Seventieth Annual General Meeting and Annual Dinner conveyed the assurance: *In a nutshell, the Society is in very good heart.* This is true of the Society also in this seventy-first year, although the Society has suffered a grievous loss by the death of Frederic Leathley Goodall, who so actively devoted himself to the duties of President.

The then President-elect, Mr. Fred Smith, responded to the call made upon him by the Council of the Society, who unanimously elected him to the Presidency, to carry on the onerous duties of his late predecessor. He was also unanimously elected Chairman of Council, and is actively furthering the interests of the Society.

Subsequently, at an Extraordinary General Meeting of the Society, Mr. Clifford Paine, then Chairman, Dyestuffs Division, Imperial Chemical Industries Limited, was unanimously elected President-elect in succession to Mr. Fred Smith.

The records of new members, re-admissions, resignations, and deaths show a net increase in the membership of 153.

The loss by death of thirteen other members has been reported during the year, including Mr. G. H. Schuler (Chairman, A.A.T.C.C. Publicity Committee) and Mr. F. Wilkinson (son of a Founder Member).

In the course of a very progressive year of great activity the Council is pleased to report that the Diplomas Scheme has now become operative. The Fellowship of the Society has been awarded to 114 members at home and overseas. The Council extends its congratulations to these members.

The 1954 examinations for the Associateship were held in Bradford and Galashiels on 11, 12, and 13 November 1954, and resulted in five candidates subsequently being elected Associates. The Council congratulates the successful candidates, who have thus passed the first examination to be held under the auspices of the Society, and welcomes them as Associates of the Society.

The Council has pleasure in reporting that continued good relations have been maintained with the affiliated Society of Dyers and Colourists of Australia and the Dyers and Colourists Association of South Africa. In addition, the cordial co-operation between the Society and the American Association of Textile Chemists and Colorists has continued.

The international character of the Society's activities is demonstrated by its growing overseas membership and by the leading part it has played in such events as the third meeting of the International Standards Organisation Colour Fastness Subcommittee, which was held under the auspices of the British Standards Institution and the Society, acting through the Fastness Tests Co-ordinating Committee. On this occasion fourteen countries were represented.

The formal opening of the rebuilt research laboratory of the Naphtol Chemie Offenbach works of the Farbwerke Hoechst AG. in Germany,

to be known as the *Laska–Zitscher Laboratory*, was attended by the President, Mr. Fred Smith, and other representatives of the Society, who made a special journey in tribute to Dr. A. Zitscher. Subsequently, Dr. Zitscher was honoured by the Society with the award of the Perkin Medal for his work leading to the discovery of the new class of azoic dyes, based on the arylamides of *o*-hydroxycarboxylic acids.

At home, the Society continues to have cordial relations with cognate organisations. In this respect, under the joint auspices of the Society and the Textile Institute, a very successful Conference on *Colour and Textiles* was held at Scarborough, 8–11 June 1954, the proceedings of which were subsequently published jointly.

The Society is honoured by its helpful relationship in the organisation of the Perkin Centenary celebrations with the Royal Society, the Chemical Society, the Society of Chemical Industry, the Royal Institute of Chemistry, and the Association of British Chemical Manufacturers. The Council extends thanks to the Dyers and Finishers' Association for the use of the Association's Board Room for a period during the year. Further, the Council wishes to place on record its gratitude to the Association for the gift of an annual prize of ten guineas for competition between Junior Members to be allocated by the West Riding Section Committee.

During the year Council extended congratulations to *The Dyer* on celebrating its seventy-fifth anniversary. This publication played an important part in the founding of our Society, for its then manager, Mr. S. J. Sewell, convened the inaugural meeting of the Society in 1884.

The Society's Second Annual Golf Competition was held on 30 May 1954 on the course of the Cavendish Club, Buxton, when there were eighteen competitors. The occasion was a great success.

Council is indebted to the authors of papers and communications, abstractors, and to all who co-operate in making the *Journal* the success it is.

The advertisers in the *Journal* have increased and Council records gratitude for this greatly valued support of the *Journal*.

The Council has received with pleasure annual donations, and thanks those concerned for these generous gifts.

The activities of the Sections and Junior Branches continue to gather momentum, the interest of members being attracted by means of lectures, film shows, and social functions. Council is convinced that all functions of this kind play an important part in the life of the Society, and thanks those who contribute to their success.

Finance Committee—The Committee have met regularly during the year. The Committee is thanked by Council for the successful year recorded in the 1954 accounts, and for the large amount of work it has devoted both to the accounts and to the business arrangements for the Second Edition of the *Colour Index*.

Society's Medals Committee—On the recommendation of the Committee, the President submitted to Council the names of C. M. Whittaker, Esq., D.Sc., F.T.I., F.S.D.C., and F. Scholefield, Esq., M.Sc., F.R.I.C., F.T.I., F.S.D.C., for the award of Honorary Membership of the Society in recognition of sustained and outstanding services to the Society. Other recommendations were the award of the Gold Medal of the Society to G. S. J. White, Esq., M.A., F.T.I., F.S.D.C., and the award of the Silver Medal of the Society to C. C. Wilcock, Esq., A.R.T.C. (Salford), F.T.I., F.S.D.C., both for exceptional services to the Society and to the tinctorial and allied industries. All four recommendations were unanimously accepted by Council.

Worshipful Company of Dyers Research Medal Committee—The Council welcomed the Committee's recommendation that the Research Medal be awarded to Mr. Ramsay J. Hannay for his work on pH control in dyeing, and this recommendation was subsequently acceptable to the Court of the Worshipful Company of Dyers.

Worshipful Company of Feltmakers Research Medal Committee—The Council and the Adjudication Committee regret that it has not been possible to recommend an award, but they hope that this position will be remedied in the near future.

Perkin Centenary Organising Committee—Sir William Perkin died when in the office of President of the Society, and only last June the Society was bereaved of a President, the late Mr. F. L. Goodall, who had very much at heart the efforts to honour the founder of the Synthetic Dye Industry.

Someone had to start up the machinery, and the Council feels that all honour is due to the late Mr. Goodall and to those who supported him in his very successful efforts to initiate interest.

The President of the Society came to be recognised as the nominal head of the proposed Centenary Celebrations, and the onerous duties arising therefrom have been carried on by Mr. Fred Smith.

The Council, through its Perkin Centenary Organising Committee, responded to the wishes of the kindred bodies who had been approached to join with the Society in sponsoring the Celebrations, and the Society's own Committee has become absorbed in the Committees now representative of the six sponsoring bodies.

The Council is deeply gratified that the approaches made by the Society culminated in the great honour of H.R.H. The Duke of Edinburgh consenting to become Patron of the Perkin Centenary.

Symposium—The Council decided in favour of holding the next Symposium in Northern Ireland and this will be held on 14, 15, and 16 September 1955 at Portrush. A representative Committee was set up to complete arrangements. These now include an interesting programme for the many visitors the Council hopes will be attracted to this first Symposium to be held in Northern Ireland. The title will be *Bleaching, Dyeing, and Finishing Today*.

MERCER LECTURE—The responsibility for delivering the Eleventh Mercer Lecture, on *Physics in the Dyeing, Printing, and Finishing Industries*, was placed in the hands of Mr. N. W. Yelland, the chair being taken by the late Mr. F. L. Goodall, the then President. The meeting was very well attended, and Mr. Yelland's valuable contribution was subsequently published in the Society's *Journal*.

The twelfth lecture has been entrusted to Dr. G. Landells, who will speak on *Modern Resin Finishing* at the Midland Hotel, Bradford, on Thursday, 17 March 1955.

The Mercer Lecture is now recognised as a very important permanent feature of the Society's activities, and the Committee has considered the best methods of enhancing the success and general popularity of the twelfth lecture of the series. The policy is continued of inviting lecturers from among the younger research workers, chemists, and technologists to provide them with opportunities of addressing members of the Society and other persons interested in textile processing.

GEORGE DOUGLAS LECTURE—At the suggestion of the Committee the Council of the Society agreed that the next Lecture should take place in the Perkin Centenary Year 1956.

PUBLICATIONS COMMITTEE—The standard of the *Journal* continues at a high level. During 1954 the number of lectures and communications has increased by 25% compared with the number published in 1953 other than the papers given at the Symposium on Textile Printing. Abnormal arrears of publication have now been overcome, and both papers and abstracts are being published without undue delay. The Committee again wishes to record its appreciation of the generosity of those firms which enable expensive colour reproductions to be included in certain scientific papers.

The Publications Committee has devoted considerable thought to how best members and other readers of the *Journal* who have difficulty in finding time to keep abreast of scientific developments may be assisted to understand modern theories. The Committee has decided to publish a series of papers which will summarise, as simply as is possible, scientific advances which are of interest to practical dyers. This series is to begin in 1955, and the Committee is grateful to the contributors, who have attempted a most difficult task. The Committee trusts that a closer understanding between theory and practice, for the general benefit of the technology of coloration, will be thereby achieved, and it will welcome comments from members.

The Committee is giving serious attention to remedying the dearth of authoritative monographs and textbooks in the field of interest of the Society, and expects to be able to report definite progress during 1955.

The Committee has been strengthened during the past year by the addition of four new members and they have already made valuable suggestions relating to the general work of the Committee.

LIBRARY—Interest in the Library has been maintained, and the number of loans has continued on the same level as in 1953. Several requests for loans have come from outside bodies, such as Government Departments and firms, stimulated no doubt by mention of the Society's holdings of periodicals in the *World List of Scientific Periodicals*.

Owing to difficulties of accommodation, the Publications Committee has decided to cut down drastically the number of periodicals to be retained permanently by the Society. Only those directly concerned with tinctorial technology or those not readily available elsewhere, e.g. Russian and Japanese journals, are to be retained, while other periodicals received for abstracting purposes will be kept for about a year and then disposed of.

It may again be stressed that most of the books reviewed and many of the periodicals abstracted in the *Journal* are available for loan to members of the Society.

HISTORICAL RECORDS COMMITTEE—During the year additions have been made to the Society's collection of historical documents, a display of which was given in Leicester. Much propaganda and preparatory work have been done, some of which is already bearing fruit.

DIPLOMAS COMMITTEE—This year has seen the inception of the Society's Diplomas Scheme, the election of the first Fellows, and, most important of all, the holding of the first examinations for the Associateship. A vast amount of work has been done, mostly by members of the Diplomas Executive Subcommittee, and it is confidently expected that this has laid the foundations of a smoothly working scheme which will be of immense benefit to our younger members and those entering the tinctorial industries. Several teaching institutions have already commenced special courses to prepare candidates for the Society's examinations, and in that and in other directions it is clear that the introduction of the Society's Diplomas is having a beneficial effect on the education of those training to become tinctorial technologists.

FASTNESS TESTS CO-ORDINATING COMMITTEE—The Committee held six meetings during the year and has continued its activities toward internationally accepted tests for colour fastness.

The highlight of the year was the third meeting of the Colour Fastness Subcommittee of the International Organisation for Standardisation (ISO/TC 38/SC 1) held at Scarborough in June. Ten members of the Society were present, and final agreement on twenty-four tests was reached. After the necessary but purely formal ratification by the main Technical Committee, these tests should eventually be established as national standards in each of the thirty-three member countries. These tests, listed in J.S.D.C., **70**, 294 (1954), are to be published almost simultaneously in Europe and probably in the U.S.A. in May or June 1955. Publication in the *Journal* will include also those tests which are still being considered by I.S.O. together with the residue of tests published in the *Second Report of the Fastness Tests Committee* which are still valid.

Apart from the I.S.O. Conference, close contact has continued with the European Fastness Commission (E.C.E.), the chairman attending a committee meeting in Düsseldorf in November.

The technical subcommittees have continued their work, and the Perspiration and Burnt Gas Fumes Subcommittees have developed revised testing methods which are now being considered by I.S.O. The Light Fastness Subcommittee has been particularly active during the year: the daylight test (*B.S.* 1006 : 1953) having been accepted with minor modifications by I.S.O. (the amended version being *B.S.* 1006 : 1955), the subcommittee began an extensive investigation into the use of fading lamps with the ultimate object of devising a standard testing method. The Washing Fastness Subcommittee met on four occasions during 1954 and made a substantial contribution towards establishing fastness tests for cellulosic and animal fibres acceptable to the I.S.O.

The Committee has sponsored two publications in the *Journal*, one dealing with the continuing investigation into the development of a more reliable perspiration test, and the other reviewing the possible causes of discrepancies which sometimes occur when fading lamps are used and indicating the lines on which the Light Fastness Subcommittee should proceed.

Textile Institute Committee "D"— Four meetings have been held during 1954, the main topics under discussion being— (1) Methods of fluidity measurements for cotton and viscose and acetate rayons; standard procedures were finally evolved. (2) Removal of added matter from loom-state or finished cloths. (3) Analysis of viscose rayon and cotton mixtures. All these have entailed an appreciable amount of laboratory work, but item (3) is still not finalised.

Representation on B.S.I. Committees— *Rayon Industry Committee*— Consideration has been given to draft specifications for furnishing fabrics. These specifications were put forward by the Furnishing Fabrics Federation and deal with both cotton and cotton and rayon mixtures. No final conclusions have been reached and further specifications are being prepared.

The Textile Divisional Council is considering the advisability of prescribing details of the process and procedure to be used in the dry cleaning of uniforms following a suggestion by the Local Authorities Standards Advisory Committee, because supplies officers who were responsible for large-scale dry cleaning contracts found considerable divergence in practice and consequently in prices quoted. A draft was prepared with the approval of the National Federation of Dry Cleaners, and resulted in the publication of *B.S.* 2514.

Wool Industry Committee— Three meetings have been held and a variety of items have been discussed. The Committee looks to the Society for help in problems of colour fastness of textile materials.

Linen Industry Standards Committee— The annual meeting of this Committee was held at Portstewart, Northern Ireland, on 8th April 1954. As a means of fixing standards of sizes of towels, sheets, etc., and for getting Army, Air-Force, and Navy requirements standardised instead of all different as they formerly were, this Committee does fulfil a desirable function.

Subcommittee CHE/18/3— Standards for Anodic Oxidation Finishes for Aluminium and Aluminium Alloys— The Subcommittee met on three occasions in 1954, for consideration of comments received on Draft British Standard for Anodised Aluminium, Revision of *B.S.*1615. An investigation is to be started in an attempt to express a specific abrasion resistance.

Regional Advisory Council for Technical and Other Forms of Further Education for Manchester and District— One meeting has been held, at which it was agreed to hold courses on the Physical and Chemical bases of Fibre Technology and on Chemical Finishes at the Manchester College of Technology. In addition, one other course at Manchester and three courses at the Royal Technical College at Salford, on Textile Manufacturing processes, were suggested.

Terms and Definitions Committee— Meetings of the Terms and Definitions Committee were held in May and June 1954, at which a further twenty-eight terms were defined. At the June meeting it was considered that the Committee had reached the end of its immediate remit and that the remaining terms should be published in the *Journal* for comment, after which the Committee would meet again and revise all definitions in the light of criticism received with a view to the publication of a completed glossary. Unfortunately, the last set of definitions were not accepted by Council, and a further meeting had to be held in January 1955 to modify these terms and ratify the minutes. Publication of the terms is awaited.

Colour Index Editorial Panel— During the year the work has advanced to the stage where the first proofs are available of the main bulk of Parts I and II. These first proofs have been submitted to the co-operating dye manufacturers and also to the A.A.T.C.C. This has been a prodigious task for all concerned, and the Panel is greatly indebted to the co-operating dye manufacturers for the manner in which these proofs have been dealt with. This stage of the work has taken longer than was anticipated, which must be accepted as inevitable in the interests of accuracy.

The production timetable has, of course, had to be adjusted in accordance with the progress of the work of revision, and it is now recognised that publication cannot take place before early 1956.

Close and cordial liaison with the *Colour Index* Committee of the A.A.T.C.C. has been maintained, and many problems relating to both the technical and the business side of the production have been satisfactorily resolved. The work of the Panel continues to be extremely onerous, and Council records its indebtedness to members for their endeavours and also to the firms and institutions who have allowed members of their staffs to take part in this work. Mention must also be made of the invaluable co-operation given by the printers,

Messrs. Chorley & Pickersgill Ltd. and Messrs. Lund Humphries & Co. Ltd.

PARLIAMENTARY AND SCIENTIFIC COMMITTEE—The Committee comprises 161 Peers and Members of Parliament and the representatives of 94 Scientific and Technological Institutions, including the Society. It is a non-party body formed with the object of providing a permanent liaison between scientific bodies and Parliament as well as a centre for the consideration and discussion of scientific information bearing on current proceedings in Parliament. The work of the Committee forms an independent Report, which is available on loan from the Offices of the Society.

ROYAL SOCIETY NATIONAL COMMITTEE FOR CHEMISTRY—Two meetings have been held, these being principally concerned with arrangements for international conferences.

ROYAL TECHNICAL COLLEGE, SALFORD—APPLIED SCIENCES ADVISORY COMMITTEE—Two committee meetings per year are held and last year there was a special meeting. At one time this institution provided excellent part-time tuition in Textile Chemistry. Before and during the war these resources and facilities formerly devoted to the training of dyers were devoted to other purposes, but new facilities are being provided in the new building extensions. Contact with a group from the local Textile Finishing Trades to act as an Advisory Committee has been successful. A meeting is planned of the whole Textile Finishing trade as soon as it is convenient, in order to review the relationship of the Salford Royal Technical College with the trade.

COMMITTEE ON METHODS FOR MEASUREMENT OF DYE AFFINITY OF REGENERATED CELLULOSE—The Committee reported their conclusions to the Council and advocated that their further functioning terminate since an absolute method of measuring dye affinity appears to involve such rigid control as to make it impracticable for routine use. Furthermore, the lack of a suitable cellulosic material of standard dye affinity precludes the use of a comparative method. In accepting this position the Council paid tribute to the excellent work the Committee had done in seeking to find measurement methods. The Council extends its best thanks to the Committee and to all concerned with their efforts in relation to an ever recurring problem.

The Council concludes this report with gratitude to all members who have rendered service to the Society in an honorary capacity and to firms and institutions for their co-operation in making this possible.

The administration of the Society has been admirably led by the General Secretary, Mr. J. W. Nicholls, and the *Journal* has maintained its usual high standard under the Editorship of Dr. C. J. W. Hooper, whose services as Technical Officer are also appreciated.

Finally, the Council is appreciative of the excellent work done by the members of the staff throughout a year when the activities have been particularly onerous, owing to the further expansion of the Society.

In presenting and moving the adoption of the Annual Report of Council, the Honorary Secretary (Mr. J. G. HOPKINSON) supported the President's reference to the tragic loss of Mr. F. L. Goodall during his term of office as President.

Mr. Hopkinson paid tribute to Mr. Fred Smith for assuming the Presidency at short notice and referred to Mr. Clifford Paine's acceptance of nomination and election to the office of President-elect.

As regards the Annual Report, he desired to draw attention to a few items of outstanding interest—

The Diplomas Scheme—First examinations had been held and the success of the diplomas scheme for the future was hoped for.

The Perkin Centenary was occupying a large part of the Society's outlook.

The Second Edition of the "Colour Index" was another big enterprise in the Society's schemes and publishing was being expedited.

The Golf Competition was a great success last year, and attention was invited to the 1955 competition already announced.

The Mercer Lecture continued to attract growing interest, for, not only had the 1954 event in Manchester been a great success, but in 1955 there had been an attendance of over 300 when the Lecture was held in Bradford.

The Ballot for Vice-presidents and Ordinary Members of Council had received the most careful attention of the scrutineers, to whom he extended thanks for their valuable services.

In conclusion, Mr. Hopkinson recorded his very great regret that for business reasons he must relinquish the office of Honorary Secretary, and he expressed his good wishes to his successor.

Mr. S. R. MEADOWS seconded the adoption of the Report, and in so doing paid warm tribute to the work of Mr. Hopkinson and expressed the deep regret felt by everyone that he was not able to continue as Honorary Secretary.

Mr. H. À BRASSARD drew attention to the format of the Report and Accounts, which he regarded as uninteresting and not conveying the importance and dignity of the Society as did those of other societies.

The President assured Mr. à Brassard that his observations would be noted for reference to the Council.

On a show of hands the adoption of the Report was declared carried unanimously.

ANNUAL ACCOUNTS

The Honorary Treasurer (Mr. H. JENNISON), in presenting the accounts for the year ended 31st December 1954, referred to the surplus of £3,438 4s. 6d., and explained various aspects of the Accounts. He mentioned the mixed honour of initially financing the Perkin Centenary celebrations, for most of which expenditure, he was assured, the Society would be reimbursed as soon as funds were available.

Balance Sheet as at 31 December 1954

31.12.53		SURPLUS AND LIABILITIES	£ s. d.	£ s. d.
		Accumulated Funds Account		
30,163		Accumulated Funds at 1.1.54	33,519 16 11	
		Add —Library Introduced as Asset ...	100 0 0	
3,337	33,620	Surplus for the year ...	3,438 4 6	37,058 1 5
	1,851	**Foundation Fund** ...		1,850 13 0
	100	**Knecht Memorial Fund**		100 0 0
		George Douglas		
2,000		**Lecture Fund**... ...	2,000 0 0	
		George Douglas		
151	2,151	**Accumulated Income**	208 14 6	2,208 14 6
		Colour Index (2nd Edition) Prepayments		
	12,746	**Scheme**		14,664 0 7
	162	**Subscriptions Received in Advance** ...		121 13 1
		Sundry Creditors and Provisions for Miscellaneous Expenditure (Per		
	22,116	Schedule)		9,328 15 1
		Colour Index (2nd Edition)		
		Note— At the date of the Balance Sheet there may exist a liability in respect of a proportion of expenditure incurred by the American Association of Textile Chemists and Colorists of which half is payable by the Society, which is not ascertainable at the date of this Balance Sheet. Provision for an estimated liability is incorporated in the accounts.		
		H. JENNISON - *Honorary Treasurer*		
		J. G. HOPKINSON - *Honorary Secretary*		
	£62,646			£65,331 17 8

31.12.53		ASSETS	£ s. d.	£ s. d.
		Accumulated Funds		
		Investments Account		
	23,165	Investments at Cost ... (Market Value £24,163)		25,329 6 4
		Foundation Fund		
	1,851	Investments at Cost ... (Market Value £1,846)		1,850 13 0
		Knecht Memorial Fund		
	100	Investments at Cost ... (Market Value £89)		100 0 0
		George Douglas Lecture Fund		
	2,000	Investments at Cost ... (Market Value £1,736)		2,000 0 0
31		**Cash in Hand**	68 13 8	
1,423		**Cash at Bank**	2,034 16 2	
4,462		**Debtors and Prepayments (per schedule)**	2,560 1 8	
1,916	7,832	**Stocks (per schedule)** ...	1,849 7 4	6,512 18 10
		Colour Index (2nd Edition) Account		
1,931		Cash at Bank... ...	1,406 6 11	
		Cash at Bank		
11,038		(Deposit Account) ...	6,093 3 6	
4,669		Stock of Paper... ...	4,668 10 0	
2,975		Work in Progress ...	1,047 0 0	
		Expenditure to date (Per Accumulations		
7,285	27,198	Account)	14,552 16 6	28,368 1 11
		Northern Ireland Symposium Account		45 9 10
		Perkin Centenary Account		305 7 9
		Furniture and Equipment		
58		Library	83 0 0	
354		General	513 0 0	
		New Colour Index (2nd		
99	500	Edition)	124 0 0	720 0 0
		Library (At Nominal Valuation)		100 0 0
	£62,646			£65,331 17 8

AUDITORS' REPORT

We have examined the above Balance Sheet with the Books and Documents of the Society produced to us. In our opinion the above Balance Sheet is properly drawn up so as to exhibit a true and fair view of the state of the Society's affairs according to the best of our information and the explanations given to us and as shown by the Books of the Society.

Bradford
9 March 1955

RAWLINSON, GREAVES & MITCHELL
Chartered Accountants
Auditors

COLOUR INDEX (SECOND EDITION) ACCOUNT

31.12.53			£ s. d.	£ s. d.
4,669		**Stock of Paper 1.1.54** ...		4,668 10 0
		Work in Progress 1.1.54		2,975 0 0
		Expenses		
		Wages and National Health		
403		Insurance	800 0 0	
2,975		Printing and Publishing ...	4,131 8 11	
		Printing, Stationery,		
380		Advertising and Postages	221 9 0	
36		Telephone	35 0 0	
11		Electricity and Heating ...	15 0 0	
17		Storage and Sundries ...	16 0 0	
		Rent, Rates and		
67		Insurance	104 0 0	
30		Audit and Accountancy ...	30 0 0	
2		Bank Charges	7 6 3	
51		Insurance on Paper ...	70 10 0	
	3,978	Repairs and Renewals ...	43 9 3	5,474 3 5
	£8,647			£13,117 13 5

31.12.53		£ s. d.
4,669	**Stock of Paper 31.12.54**	4,668 10 0
2,975	**Work in Progress 31.12.54**	1,047 0 0
	Investment Income	
113	Midland Bank Ltd. Deposit Account... ...	134 17 0
	Balance carried to Colour Index	
890	Accumulations Account	7,267 6 5
£8,647		£13,117 13 5

COLOUR INDEX (SECOND EDITION) ACCUMULATIONS ACCOUNT

		£ s. d.
6,395	**Accumulated Total at 31.12.53**	7,285 10 1
890	**Balance** brought forward being Excess of Expenditure over Income for the year ...	7,267 6 5
£7,285		£14,552 16 6

		£ s. d.
7,285	**Accumulated Total at 31.12.54**	14,552 16 6
£7,285		£14,552 16 6

CONSOLIDATED GENERAL REVENUE ACCOUNT for the Year ending 31 December 1954

31.12.53		EXPENDITURE	£ s. d.	£ s. d.
		General Expenses		
332		Printing and Stationery	275 1 4	
142		Postages	78 15 5	
1,674		Salaries, Wages and Superannuation and Staff Expenses, etc.	1,362 18 6	
290		Rent, Rates and Insurance	251 2 5	
43		Electricity and Heating	46 19 4	
65		Telephone	60 0 0	
21		Bank Charges	19 10 0	
167		Audit and Accountancy	150 0 0	
84		Travelling Expenses ...	135 7 11	
22		Repairs and Renewals	82 10 11	
164		Miscellaneous Committee Expenses	124 7 8	
479		Sundries	462 11 8	
4	3,433	Investment Charges (Midland Bank Executor and Trustee Co. Ltd.)	6 19 6	3,007 5 8
		Scarborough Conference		327 10 3
	773	**Sectional Expenses** ...		802 15 8
	46	**Depreciation of Office Furniture and Equipment**		33 0 0
		Mercer Lecture		
57		**Expenses**	67 10 11	
20	37	*Less*— Donation ...	20 0 0	47 10 11
		Journal		
530		Stock— 1st January 1954	550 0 0	
8,647		Printing and Publishing	7,643 2 1	
682		Postages	628 13 0	
1,194		Salaries, Honoraria and Wages	2,165 6 0	
156		Own Journals	21 2 4	
		Sundries	209 13 4	
296		Abstractors' Fees and Book Reviews ...	402 3 11	
		Miscellaneous Committee Expenses	57 8 8	
17		Miscellaneous Publications	25 7 6	
		Repairs	81 0 0	
82		Rent, Rates and Insurance	157 10 0	
18		Audit	50 0 0	
		Electricity and Heating	40 0 0	
30		Depreciation	34 7 6	
		Telephone	52 12 0	
		Travelling Expenses ...	60 0 0	
104	11,606	Printing and Stationery	104 0 0	12,232 6 4
		Annual Meeting and Dinner		
60		Meeting	62 19 7	
51	111	Dinner	121 16 1	184 15 8
	33	**Donations and Subscriptions** ...		36 17 0
		Unpaid Accounts		
	89	Provision for 1954 ...		250 0 0
		Previous years' now written off (less provisions in respect of those years)		494 9 7
	54	**Library Upkeep** ...		12 19 3
		Printing of Bye-laws ...		14 0 0
		Colour Index (2nd Edition) Expenses incurred but not allowable per agreement with A.A.T.C.C		95 14 3
	46			
		Fastness Tests Account		
1,510		Stock 1st January 1954	514 17 4	
274		Materials Purchased ...	2 18 9	
14		Postages	5 2 2	
37		Loose Patterns and Bunches	79 10 0	
	1,835	Sundry Committee Expenses	50 15 4	653 3 7
	18093			18,192 8 2
	3,336	**Surplus for the Twelve Months**		3,438 4 6
	£21,429			£21,630 12 8

31.12.53		INCOME	£ s. d.	£ s. d.
		Subscriptions		
6,686		Members	7,100 6 10	
64	6,750	Juniors	115 8 0	7,215 14 10
		Journal		
		Sale of Journals, Reprints and Combined		
4,771		Reports, etc. ...	4,073 19 4	
6,397		Advertisements ...	7,683 10 9	
		Stock— 31st December		
550	12,218	1954	550 0 0	12,307 10 1
		Investment Income (Gross)		
33		Foundation Fund ...	33 16 8	
597	630	Development Fund ...	631 14 3	665 10 11
		Profit on Sale of Investments		7 3 4
	153	**Donations**		165 15 0
		Fastness Tests Account		
204		Grey Scales	81 19 3	
154		Staining Scales ...	11 18 1	
28		Reports	17 0 8	
171		Materials	235 8 0	
497		Bunches and Loose Patterns	262 10 0	
515	1,569	Stock— 31st December 1954	333 15 9	942 11 0
	110	**Surplus on Sales of Publications** ...		226 7 6
	£21,429			£21,630 12 8

GEORGE DOUGLAS ACCUMULATION FUND ACCOUNT

		£ s. d.
57	**Lecture Expenses**	– – –
2	**Bank Charges**	2 0 0
151	**Balance** per Accumulations and Appropriations Account 31.12.54...	208 14 6
£210		£210 14 6

		£ s. d.
151	**Balance** per Accumulations and Appropriations Account 31.12.53...	151 4 4
59	**Investment Income (Gross)** 3% British Transport Stock 1978/88	59 10 2
£210		£210 14 6

SCHEDULE OF DEVELOPMENT ACCOUNT INVESTMENTS

	Balance 1953	*Alterations*	*Balance 1954*	*Middle Market Value 1953*	*Middle Market Value 1954*	*Middle Market Quotation*
	£ s. d.		£ s. d.	£ s. d.	£ s. d.	
£2,763 3s. 8d. 3% British Transport Guaranteed Stock 1978/88	*2,593 17 0*		2,593 17 0	*2,326 5 1*	2,417 15 7	87¼
£750 2½% Funding Stock 1956/61	*750 0 0*		750 0 0	*734 1 3*	749 1 3	99¾
£546 3½% Conversion Loan	*557 0 0*		557 0 0	*465 9 5*	482 10 7	88¼
£1,255 2½% Defence Bonds	*1,255 0 0*	£1,000 sold during year and re-invested in Treasury 3½% Stock 1979/81 (Profit on sale £7 3s. 4d.)	255 0 0	*1,255 0 0*	255 0 0	—
£2,749 16s. 0d. 3% Savings Bonds 1960/70... ...	*2,750 0 0*		2,750 0 0	*2,509 3 10*	2,631 4 4	95⅝
£750 2½% Exchequer Stock 1955	*750 0 0*		750 0 0	*749 10 3*	750 4 8	100⅛
500 National Savings Certificates 15s. Units 7th Issue	*375 0 0*		375 0 0	*375 0 0*	375 0 0	—
£2,000 3% British Gas Stock 1990/95	*2,000 0 0*		2,000 0 0	*1,795 0 0*	1,750 0 0	87½
£2,017 11s. 6d. 3% Treasury Stock 1966 or after ...	*1,957 1 0*		1,957 1 0	*1,505 12 3*	1,563 12 5	77½
£901 3s. 6d. 3% Southern Rhodesian 1971/73 ...	*910 13 9*		910 13 9	*770 10 1*	815 11 2	90½
York County Savings Bank	*3,213 3 8*	£780 Deposited during year plus £87 2s. Interest and 12s. 2d. Interest on Defence Bonds	4,080 17 10	*3,213 3 8*	4,080 17 10	—
£1,500 12s. 9d. 3% British Electricity Guaranteed Stock 1968/73	*1,495 19 0*		1,495 19 0	*1,338 7 8*	1,418 2 5	94½
Post Office Savings Bank	*530 15 2*	£500 Deposited during year plus £18 19s. 6d. Interest	1,049 14 8	*530 15 2*	1,049 14 8	—
£1,000 Chesterfield Corporation 3% Stock 1960 ...	*993 15 0*		993 15 0	*985 0 0*	1,000 0 0	100
Midland Bank Ltd. Deposit Account	*3,028 5 6*	£500 Deposited during year plus £49 19s. 3d. Interest	3,578 4 9	*3,028 5 3*	3,578 4 9	—
£1,038 Treasury 3½% Stock 1979/81		Purchased during year	1,007 3 4		1,025 13 6	98⅝
£220 3½% Defence Bonds		Purchased during year	220 0 0		220 0 0	—
	£23,165 10 1		£25,329 6 4	*£21,491 3 11*	£24,162 13 2	

SCHEDULE OF FOUNDATION FUND INVESTMENTS

	£ s. d.		£ s. d.	£ s. d.	£ s. d.	
£1,245 2½% Defence Bonds	*1,245 0 0*		1,245 0 0	*1,245 0 0*	1,245 0 0	—
1,000 National Savings Certificates 10s. Units 8th Issue	*500 0 0*		500 0 0	*500 0 0*	500 0 0	—
£68 8s. 6d. 3% Savings Bonds 1960/70	*69 9 0*		69 9 0	*62 3 9*	65 12 11	95⅝
Midland Bank Ltd. (Cash awaiting Investment) No. 2 Account	*36 4 0*	£36 4s. 0d. Withdrawn during year and Invested	36 4 0	*36 4 0*	35 15 2	98⅝
£37 6s. 6d. Treasury 3½% Stock 1979/81						
	£1,850 13 0		£1,850 13 0	*£1,843 12 9*	£1,846 8 1	

GEORGE DOUGLAS LECTURE FUND

£1,983 17s. 3% British Transport Stock 1978/88 ...	*£2,000 0 0*	£2,000 0 0	*£1,670 3 4*	£1,735 17 4	87½

KNECHT MEMORIAL FUND

£98 16s. 6d. 3% Southern Rhodesia 1971/73 ...	*£100 0 0*	£100 0 0	*£84 9 11*	£89 8 9	90½

SCHEDULE OF DEBTORS AND PREPAYMENTS

	31.12.53	31.12.54 (*Approximate Analysis*)
	£ s. d.	£ s. d.
Advertisements...	*2,145 15 8*	1,412 4 7
Journals, Reprints, and Combined Reports...	*1,525 16 8*	468 2 7
Fastness Tests, etc.	*191 9 0*	93 15 0
Miscellaneous Publications, Small Accounts, etc.	*243 9 7*	166 9 4
Rates and Insurance Paid in advance ...	*98 16 3*	59 15 1
Income Tax repayment due	*96 5 3*	104 15 1
Subscriptions and Donations	*150 0 0*	150 0 0
Cost of Arms for the Society		105 0 0
	£4,461 12 10	£2,560 1 8

SCHEDULE OF CREDITORS AND PROVISIONS FOR MISCELLANEOUS EXPENDITURE

	31.12.53	31.12.54
	£ s. d.	£ s. d.
Printing and Stationery	*51 16 0*	184 16 5
Accountancy and Professional Charges ...	*170 0 0*	200 0 0
General Expenses	*29 19 4*	48 1 6
Sectional Expenses	*39 14 8*	96 19 2
Journal Expenses—		
Printing and Publishing	*3,782 11 7*	2,545 19 5
Postages	*173 9 10*	194 5 6
Amounts received in Advance	*76 19 6*	121 13 1
Miscellaneous Committees	*15 17 11*	73 10 5
Royalties *Wool Dyeing*	*17 3 6*	30 0 0
Printing *Symposium*	*153 8 8*	
Scarborough Conference Expenses		10 0 0
Book Reviews and Abstracting		95 10 2
Credit balances in Sales Ledger		102 12 6
	£4,473 1 0	£3,703 8 2
COLOUR INDEX (2nd Edition)		
Printing and Publishing	*2,975 0 0*	1,047 0 0
General		31 10 0
A.A.T.C.C. (Paper Supplied)	*4,668 10 0*	4,668 10 0
	£12,116 11 0	£9,450 8 2

SUMMARY OF STOCK

	1953	1954
	£ s. d.	£ s. d.
Fastness Tests and Materials	*514 17 4*	333 15 0
Journals	*550 0 0*	550 0 0
Photochemistry Symposium	*12 16 0*	10 0 0
Replika *Colour Index* and Supplement ...	*50 16 0*	39 14 9
Identification of Dyes	*112 10 0*	50 0 0
Wool Dyeing	*414 0 0*	380 2 6
Stationery	*100 0 0*	200 0 0
Combined Reports, etc.	*153 5 0*	120 0 0
Tinctorial Arts Conference	*8 1 0*	6 15 0
Textile Printing Symposium		78 0 0
Scarborough Joint Conference		50 0 0
Reprints...		31 0 1
	£1,936 5 4	£1,849 7 4
Colour Index (2nd Edition)		
Stock of Paper	*4,668 10 0*	4,668 10 0
Work in Progress	*2,975 0 0*	1,047 0 0
	£7,643 10 0	£5,715 10 0

SCHEDULE OF OFFICE FURNITURE AND FITTINGS

	General		*Colour Index (2nd Edition)*		*Library*	
	1953	1954	*1953*	1954	*1953*	1954
	£ s. d.	£ s. d.	*£ s. d.*	£ s. d.	*£ s. d.*	£ s. d.
Balance 1st January...	*358 11 5*	354 0 0	*97 2 4*	90 2 4	*62 2 6*	56 0 0
Additions ...	*36 15 9*	216 15 6	*4 0 0*	47 7 7		36 12 0
	395 6 5	570 15 6	*101 2 4*	137 9 11	*62 2 6*	92 12 0
Less Depreciation @ 10% ...	*39 6 5*	57 15 6	*11 0 0*	13 9 11	*6 2 6*	9 12 0
Total per Balance Sheet	*£356 0 0*	£513 0 0	*£90 2 4*	£124 0 0	*£56 0 0*	£83 0 0

Among salient points dealt with were explanations of the *Colour Index* Accumulations Account amounting to £14,552 16s. 6d., which was concerned with the mutual liability now under consideration with the AATCC. However, it was to be hoped that next year it would not be necessary to insert a qualified statement of that nature, although it should be noted that half the prepayment subscriptions had already been spent.

The General Revenue Account on the expenditure side showed a reduction of general expenses, but this was reflected in the upward trend of *Journal* costs, due to the reallocation of charges, which, however, still showed the *Journal* to be self-supporting.

With reference to an item of £494 9s. 7d. written off in respect of unpaid accounts due to the Society, Mr. Jennison pointed out that these were balances which cleared the ledgers within a few pounds of what remained owing prior to 1954.

Mr. Jennison referred to the satisfactory state of revenue including the increased subscriptions. He further drew attention to the condensed form of the Sales of Publications, which showed the surplus resulting from these activities. In the field of investments, there had been some changes which he enumerated, and the remaining items in the Accounts appeared to be self-explanatory.

Mr. R. J. HANNAY, in proposing the adoption of the Accounts, paid tribute to the very careful work of the Finance Committee in looking after the wellbeing of the finances of the Society.

Mr. A. P. KERSHAW seconded.

Dr. C. M. WHITTAKER protested regarding inclusion of sundries in the General Expenses Account, and asked that in future more details be given. He challenged the cost of the Scarborough Conference in view of the registration fees charged, and also drew attention to the loss on the Annual Dinner, which, he contended, should be self-supporting.

Answering Dr. Whittaker regarding the amount appearing in the Accounts concerning the Scarborough Joint Conference with the Textile Institute, the Honorary Treasurer explained that £260 concerned printing of the Proceedings of the Conference for issue to Members of our Society, and £30 was for postages. The balance of £37 10s. 3d. was for travelling and hotel expenses connected with administration.

Dr. Whittaker contended there was £1,400 of "water" in the Balance Sheet, and instanced investments appearing at cost, whereas the market value was £1,400 less.

The Honorary Treasurer urged that comparisons were available on the face of the Balance Sheet which were in accordance with the procedure adopted by the Society on the advice of its Auditors.

Mr. H. À BRASSARD complained that Sundries, Sectional Expenses, and Travelling and Committee Expenses were high.

The Honorary Treasurer explained that our increasing activities were bound to be reflected in the Accounts.

Mr. H. FOSTER referred to his fifteen years as Honorary Secretary of the West Riding Section, and said that, where charges were claimed by Sections, the Society had received good value. With regard to investments, he suggested that Dr. Whittaker would still find a surplus after allowing for the difference between cost and market value.

Dr. Whittaker submitted that a realistic Balance Sheet was what was wanted.

Mr. G. A. F. MERCER wished to have Bad Debts reduced, to which the Honorary Treasurer replied that that was why nearly £500 had been written off.

The Honorary Treasurer offered to consult the Finance Committee regarding the valuation of Investments in the Balance Sheet.

The President now put the proposition to the meeting for the adoption of the Balance Sheet and Accounts for the year ended 31st December 1954, which was carried without dissent.

ELECTION OF PRESIDENT

Mr. F. SCHOLEFIELD proposed the election of Mr. Fred Smith as President. He had known him for a very long time as a friend. On the technical side he was well qualified to be President, and on the administrative and commercial side he had had many years of industrial experience.

Mr. H. FOSTER, in seconding, said that, although he had a slight seniority over Mr. Fred Smith, he could not think of anyone better suited technically and administratively to be President.

The Honorary Secretary then put the proposition to the meeting, and on a show of hands and with acclamation Mr. Fred Smith was unanimously elected President of the Society.

In responding, the President expressed his thanks and great appreciation of the continued honour accorded him. He declared that his object was always to serve every member equally.

ELECTION OF PRESIDENT-ELECT

Dr. C. M. WHITTAKER, in proposing Mr. Clifford Paine as President-elect, expressed the great pleasure he felt on this occasion. Mr. Clifford Paine would be President in the 1956 Perkin Centenary year, when he hoped the main theme would be "The Renaissance of the British Dyestuffs Industry". He was proud to have seen the British dyemaking industry become equal to that of any other country. Dr. Whittaker went on to recount experiences in the industry before the First World War with competition such as present staffs never encountered.

Dr. Whittaker spoke of the special qualifications which Mr. Clifford Paine had for the office of President of the Society, and he moved that he be now elected President-elect.

Mr. A. S. FERN expressed the great pleasure he felt in supporting and seconding the proposition submitted by Dr. Whittaker.

On a show of hands Mr. Clifford Paine was unanimously elected President-elect.

Mr. CLIFFORD PAINE, in response, expressed the assurance of his being very sensible of the honour of this office, which he hoped his thirty-six years in industry would help him to hold successfully. He reminded his hearers that he was a "dyed-in-the-wool colour chemist". He would endeavour to serve with credit in the Perkin year and to forward the interests of the Society.

RESULTS OF POSTAL BALLOT

The President announced the results of the ballot for four Vice-presidents and four Ordinary Members of Council—

*Vice-presidents**

Mr. R. J. Hannay to serve for six years

Mr. G. S. J. White to serve for three years

Mr. G. G. Hopkinson to serve for two years

Mr. W. A. Edwards to serve for one year

*Ordinary Members of Council**

Mr. A. S. Cluley

Mr. J. M. Goodall

Mr. A. Thomson

Dr. T. Vickerstaff

The Honorary Secretary reported that over 600 members had voted and over 4000 votes had been recorded.

The President, in declaring the results, congratulated the successful candidates elected as Vice-presidents and Ordinary Members of Council, and, in expressing thanks to those retiring†, he said how very much everyone appreciated their services and also the services of the scrutineers, Mr. R. Grice and Mr. J. McConville, in the counting of the votes.

ELECTION OF HONORARY TREASURER

The President proposed, Mr. J. W. Reidy seconded, and it was unanimously agreed that Mr. H. Jennison be re-elected Honorary Treasurer.

Mr. Jennison expressed his thanks and recorded his assurance of service in the best interests of the Society.

HONORARY SECRETARY

The President felt he was expressing the feelings of the Council and Members when he said how very sorry he was that, for business reasons, the services of Mr. J. G. Hopkinson must be lost to the Society.

The Council had appointed a Committee to recommend a candidate to fill the Honorary Secretaryship, and Council had subsequently nominated Mr. L. Morton Wood as Mr. Hopkinson's successor, but the election would not take place until an Extraordinary General Meeting which it was proposed to hold as soon as conveniently possible.

Dr. H. W. ELLIS rose on a point of order to enquire whether it was proposed to circulate Members notifying them of Mr. Hopkinson's resignation and of the nomination made by Council to fill the vacancy thus created. He considered it was also desirable that Members be afforded the opportunity of putting forward nominations if they so wished.

The President assured Dr. Ellis that the intention was completely in accordance with his enquiry.

APPOINTMENT OF AUDITORS

Mr. J. BARRITT proposed, Mr. N. HAMER seconded, and it was unanimously agreed that Messrs. Rawlinson, Greaves & Mitchell, Chartered Accountants, be re-appointed Auditors at a fee to be arranged.

PAST PRESIDENTS

Mr. H. À BRASSARD raised the question of the position of Past Presidents "fading away" out of the life of the Society after completion of a very valuable experience in the office of President. He advocated some form of Senate or Upper Advisory Body to which the Past Presidents might retire and thus be available for continued service to the Society.

The President assured Mr. à Brassard that this matter, having already been raised by Dr. Whittaker, was receiving the Council's careful consideration.

PERKIN CENTENARY

Mr. à Brassard pointed out that not a single member of the London Section Committee had been called upon to serve in any capacity whatsoever in connection with the Perkin Centenary Celebrations being planned for London in 1956.

The President explained that the planning of the Perkin Centenary was now directed by Committees representing the Sponsoring Societies.

VOTE OF THANKS TO CHAIRMAN

On the motion of Mr. H. JENNISON, the meeting closed with a vote of thanks to the President for presiding.

*Details of the voting are given on p. 244.

†*Retiring Vice-presidents*— Mr. G. B. Angus, Mr. C. O. Clark, Mr. H. Foster, and Professor J. B. Speakman.

Retiring Ordinary Members of Council— Mr. J. Boulton, Mr. A. Brearo, Dr. G. T. Douglas, and Mr. F. Jordinson.

Annual Dinner

The Seventy-first Annual Dinner of the Society was held at the Queens Hotel, Leeds, on Friday evening, 1st April 1955, under the presidency of Mr. Fred Smith.

The principal guest was Sir Hubert S. Houldsworth, Q.C., D.Sc., LL.D. (*Chairman of the National Coal Board and Prochancellor of the University of Leeds*). Among other guests were Mr. W. C. Castle (*Immediate Past President of the Society of Dyers and Colourists of Australia*), Mr. R. L. Runting (Honorary Secretary of the Victoria Section of the Society of Dyers and Colourists of Australia), Dr. W. E. Scott (*Principal of the Huddersfield Technical College*), Mr. R. P. Chambers (*Prime Warden of the Worshipful Company of Dyers*), Mr. D. R. Balfour Parke (*Clerk to the Court of the Worshipful Company of Dyers*), Mr. W. M. Johnson (*President of the Dyers and Finishers Association*), Mr. J. H. Shaw (*President of Bradford Textile Society*), Mr. C. G. Hulse (*Secretary of the Dyestuffs Control, Board of Trade*), Dr. A. B. D. Cassie (*Director of Research of the Wool Industries Research Association*), Mr. L. E. Morris (*Editor of "The Dyer"*), Mr. P. Caldwell, Mr. R. D. Chorley, Mr. G. H. W. Cullinan, Dr. E. Higgins, Mr. L. Morton Wood, Mr. L. E. Jones (*Honorary Patent Agent*), Mr. F. Scholefield (*Honorary Member and Past President*), Dr. C. M. Whittaker (*Honorary Member and Past President*), Mr. R. J. Hannay (*Worshipful Company of Dyers Research Medallist and a Vice-president*), Mr. C. C. Wilcock (*Silver Medallist*), and Dr. H. W. Ellis (*Chairman* of the *London Section*).

The following officers and other leading members of the Society also were present—Mr. H. H. Bowen and Dr. C. J. T. Cronshaw (*Past Presidents*); Mr. C. Paine (*President-elect*); Mr. W. A. Edwards, Mr. G. G. Hopkinson, and Mr. H. A. Turner (*Vice-presidents*); Mr. A. S. Cluley, Mr. D. A. Derrett-Smith, Mr. A. S. Fern, Mr. J. M. Goodall, Mr. N. Hamer, Mr. H. Hampson, Mr. A. P. Kershaw, Mr. J. W. Reidy, Mr. J. V. Summersgill, Mr. A. Thomson, Mr. A. G. Tyler, and Dr. T. Vickerstaff (*Ordinary Members of Council*); Mr. J. Calvert, Mr. R. K. Fourness, Mr. H. R. Hadfield, Mr. S. R. Meadows, Mr. R. A. Peel, Mr. J. Porter, Mr. R. G. J. Reid, Mr. H. W. Taylor, and Mr. H. Turner (*ex-officio Members of Council*); Mr. J. Barritt (*Chairman of the Publications Committee*); Mr. H. Jennison (*Honorary Treasurer*); and Mr. J. G. Hopkinson (*Honorary Secretary*).

The total number present was 450, a record in the history of the Society.

"THE SOCIETY OF DYERS AND COLOURISTS"

After the toast of "Her Majesty the Queen", that of "The Society of Dyers and Colourists" was proposed by—

Sir HUBERT S. HOULDSWORTH, Q.C., D.Sc., LL.D. (*Chairman of the National Coal Board and Prochancellor of the University of Leeds*), who said that he had perused several copies of the *Journal*, and found them bright as far as the advertisements went. The rest, however, was written in a language which he used to know but which his audience knew so skilfully that it was second nature to them to interpret all the formulae, so that he felt that, with his ignorance and their great knowledge, he must refrain from any reference to the *Journal* at all.

He had been told that the art and craft of dyeing was very old, older than recorded history—he hadn't the least idea how old that was, but he felt sure that everyone present would know. At any rate, next year would be the centenary of the discovery by Sir William Perkin which had transformed their art very largely into a science and which had started one of those industrial changes which in the end had greatly benefited the whole of humanity. With the typical foolishness of that day we had allowed the development to get into other hands, into those of Germany, though a belated conversion had allowed us to regain the ground which we had foolishly lost.

The production of synthetic dyes had far-reaching effects. In the nineteenth century French soldiers, who wore Turkey Red trousers, found that they had to rely for the colour of their trousers upon their enemies, the Germans, who supplied it to them. The virtual disappearance of natural indigo, due to competition from the synthetic product, released the land of the plantations for the growth of other crops, especially food.

Whatever your *Journal* may be, I am sure that your craft has given a colourful appearance to the world. You must all believe with George Bernard Shaw that, as far as the human being is concerned, it is the woman who marks down her prey and pursues it relentlessly until there is the capture and then the marriage. You have made colours for the woman: it is customary for the male to adorn himself with drabness. At any rate, I suppose that it is because of your attempt to help the female sex that you have devoted to them the best colours.

Your Society was formed twenty-eight years after Perkin's great discovery, and it has therefore been in existence some seventy-one years. I was interested to note that those who founded your Society in Bradford so long ago as that had as their object to unite scientific theory with practice in industry—how far-seeing they were! We have not learned that lesson completely even today. We have gone through a period when science, instead of indicating new lines of advance, has found scientific reasons for the practices actually being carried out—and to the credit of the empirical man has shown in most cases that these practices were sound. But real advance, whether it be in machinery, manufacture, or processing, must depend nowadays on a real uniting of scientific theory with practice. The practical man must recognise that science has its

contribution to make; but, equally, the person who is applying his scientific ideas to industry must realise that the practical man has a contribution to make to his thought. And it is only by uniting the two that the advances which are necessary will take place.

You here today fully appreciate the difficulties from which the industrial life of this country is suffering owing to the lack of technologists—owing to the lack of those who have the gift of applying scientific methods to the solution of industrial problems. We have as a nation to see that our colleges and our universities take those men who have a scientific background and a real interest in industrial processes and give them the training that is necessary to keep this country in the foreground. After all, our standard of living and our maintenance as a first-class nation must depend on our keeping ahead of our competitors in all our industrial processes. You, of course, now depend upon coal, I gather, for your raw material; and as far as your particular raw material is concerned, if my post-bag is any guide at all, you have no complaints as to quantity or quality! Indeed, the complaint is rather on our side that you have not yet learned by the appropriate application of science to assist the conservation of coal, which is so easily possible in many of the processes in your industry. Therefore I appeal to you to help me by devoting a little of your business ability to that side, as well as a little of the scientific skill of those you employ. After all, whilst coal is so cheap we shall never have higher management paying appropriate attention to its conservation unless we really show that it is necessary in the national interest.

I was interested to see that it was decided in the very first year that it was essential to have a *Journal*, and that for the past seventy years you have succeeded month by month in publishing your *Journal*—it is more than the national newspapers in London manage to do, and therefore I should like to congratulate you upon that success as well.

I wonder whether those who founded your Society could have envisaged not only its value to your whole industry in this country, but how the influence of your Society would make itself felt in other parts of the Commonwealth and in other parts of the world. I think it is a great satisfaction to any Society to have in other countries societies of a similar nature feeling some sort of kinship, as your American counterpart is concerned in combining with you in the production of the Second Edition of your *Colour Index*. The fact that your Society, in promoting interchange of ideas within an industry, is helping not only those engaged in that industry but also the whole world, must be a source of satisfaction to you all. It is because your Society has achieved so much, because so much, too, depends on the coloration of goods for home and export, and because you are striving to live up to your original object of uniting scientific theory with practice, that I welcomed the honour of coming here and proposing the toast of "The Society of Dyers and Colourists".

Mr. Fred Smith (*President of the Society*) responded to the toast and thanked Sir Hubert for the kind terms in which he had proposed it. But firstly he had the sad duty of referring to the great loss sustained by the Society in the death of his predecessor as President, Mr. F. L. Goodall.

He was very happy to be President, adorned with the badge of office so kindly presented by the Worshipful Company of Dyers, whose Prime Warden and Clerk were honoured guests that evening. The Society had for the past seventy-one years played an important part in developing the art and the science of dyeing, printing, and finishing. He might almost call it "his" Society, for he had been privileged to serve it for over forty years.

The Society was pursuing its object of encouraging education by means of its recently instituted diplomas scheme. The qualifications A.S.D.C. and F.S.D.C. were not easy to come by, for they were intended to be the hallmark of the dyer and the colourist. They would encourage the younger generation to acquire greater practical craftsmanship and greater scientific knowledge of the processes of coloration. The Society's *Journal*, which circulates all over the world, is a valuable contribution to the education of old and young alike in the tinctorial arts.

The lectures and the social activities of the Sections contributed in no small measure to the success of the Society. It was very necessary to attract young people to ensure continued health of the Society and the industry. He made a special appeal to more firms in the industry to encourage members of their staffs to take an active part in the work of the Society.

Industry was the life-blood of the nation, and both academic and practical people must realise that we cannot take out of industry more than we put into it: we must be careful not to "kill the goose which lays the golden eggs".

In the past the Society has played the rôle of "back-room boy" in the skilful application of colour, but today the value of its contribution is recognised all over the world. One of its most important functions is to foster mutual respect and co-operation between academic scientist and practical dyer, as stressed by Sir Hubert.

Finally, Mr. Smith expressed his deep appreciation of the efforts of all those who had given their time and their labour in striving for the success of the Society.

"OUR GUESTS"

Dr. H. W. Ellis (*Chairman* of the *London Section*) said that when we buy a house or acquire a factory, or marry a wife, one of the first things we do is to invite some guests to visit us. We do not do this through any desire to "show off", of course, but because of a human instinct which, somehow or other, causes us to enjoy ourselves more fully when guests are present.

We of the Society of Dyers and Colourists are therefore pleased to see, and on your behalf I am very glad to welcome, the guests whom we have

CROYDEN MEREDITH WHITTAKER
D.Sc. F.T.I. F.S.D.C.

Honorary Membership in recognition of sustained and outstanding services to the Society

FRED SCHOLEFIELD
M.Sc. F.R.I.C. F.T I. F.S.D.C.

Honorary Membership in recognition of sustained and outstanding services to the Society

GEORGE STUART JAMES WHITE
M.A. F.T.I. F.S.D.C.

Gold Medal for exceptional services to the Society and to the tinctorial and allied industries

CLIFFORD COLLIER WILCOCK
A.R.T.C. (Salford) F.T.I. F.S.D.C.

Silver Medal for exceptional services to the Society and to the tinctorial and allied industries

LASCELLES MORTON WOOD
Honorary Secretary of the Society

with us tonight. They represent collectively those who are, in various ways, interested in the "mistery of dyeing", to use a medieval phrase. Time does not permit me to refer to them severally by name, and I am sure that they will excuse me in this, but I shall permit myself one or two brief references.

You have heard our principal guest, Sir Hubert Houldsworth, propose so excellently the health of our Society. Some of you may not be aware that Sir Hubert holds the formidable distinction of being at once a Doctor of Science and a Doctor of Laws. When it is realised that, in addition, he is a Queen's Counsel, I am certain that you will agree with me that no manufacturer's representative, for example, would have the least chance of putting one across him, because what the scientist considered probable, the lawyer would have no doubt of, and the advocate would then expound to the complete discomfiture of his opponent.

Fortunately, however, for some of us, Sir Hubert does not buy dyes or other commodities. But for some time past he has been in a position which, although it is not generally recognised to be so, is of vital importance to all dye-makers and dye-users. As Chairman of the National Coal Board, he has controlled the source of supply of almost all the raw materials from which modern dyes are constructed. I mention this in case some of you are tempted to think of Sir Hubert in terms of nutty slack, and I may remind such persons that he has a much greater claim to the gratitude and the hospitality of this Society than one based upon that somewhat doubtful commodity.

Another guest whom we are particularly glad to welcome is the Prime Warden of the Worshipful Company of Dyers, Mr. R. P. Chambers. Mr. Chambers represents the oldest existing organisation connected with our craft, and is therefore assured of our acclamation and respect. It gives me particular pleasure to remind some of those present that this organisation was founded in London, and, despite the vicissitudes of history, has continued to flourish there for nearly five centuries. To those of you who are under the impression that dyeing begins and ends where roses have a political significance, I should like to quote just two sentences from a recent lecture of a rather special kind. I shall not say any more about this lecture lest I be suspected of advertising, but will simply state that the archives of the Worshipful Company of Dyers contain these noteworthy entries—In the year 1501, the Dyers' agreed with the Drapers', Tailors', and Haberdashers' Companies to acquire a Seal wherewith to mark all cloth which was London-dyed, "that it might be knowen for the best, surest and most profitable for the King's liege people of any dyeing within this Realm". Also, in the year 1482, the first ordinance of the Dyers' Company states: "No person of the craft shall rebuke or revile any other person of the same craft". While I would not go so far—I mean, while no Londoner would go so far—as to say that no other county has ever emulated their dyeing prowess, I am sure that the rest of you will agree that their standard of ethical behaviour wants some living up to.

We have with us tonight several representatives of the higher educational establishments of the country. These gentlemen will, I am sure, have noted with interest the inauguration of the Society's diplomas scheme. This is not the time to dwell upon such a matter, but I have no doubt that when, in a recent issue of our *Journal*, the text of the papers for the first Associateship Examination was published, you all, like myself, scanned them with much interest. Of course, we all regard ourselves as competent protagonists of the dyer's art and, of course, we all realise that, had we chosen to sit for the examination, we should all very easily have . . . failed!

The only others of our guests to whom I shall refer are the Honorary Members and the Honorary Secretaries of Sections. While the former are—deservedly—often in the limelight, the latter are, I am sorry to say, frequently taken for granted. Both are deserving of our thanks for their long and faithful service to our Society, but—as I am personally now able to say with propriety—particularly the Honorary Secretaries. Their work is often fairly arduous, sometimes rather boring, and always a responsibility. If all goes well, the Chairman gets the credit; but if anything goes wrong, Heaven help the Secretary! And all they often get in return is an invitation to the Annual Dinner. So I ask you all to make them especially welcome tonight.

And so, Mr. President, ladies, and gentlemen, I give you the toast of "Our Guests". May the penetration of their minds, although not the exhaustion of their wits, be always associated with the figure 5. May their resistance to adverse factors such as alteration (of fortune) or bleeding (of profits) always be maximum. May their abilities never fade, and may their courage never become unlevel at high temperatures. And lastly, may their liquor ratio never be too high for bodily equilibrium nor too low for appetite.

Mr. W. C. Castle (*Immediate Past President of the Society of Dyers and Colourists of Australia*) in response to the toast conveyed greetings from the President of the Australian Society, Mr. G. Athol Burns. Two years ago, in company with officers of the Australian Society, he had met Mr. Fred Smith, who had given a very interesting lecture, and had made it clear that he was still a practical dyer, ready at any time to take off his coat and dye a batch or two with the best. The presence of so many distinguished representatives of science and industry among the guests that evening must make Mr. Smith feel well rewarded for his more than forty years' active membership of the Society.

The affiliated Society in Australia now has a membership of approx. 350 governed by a Federal President and Council. The five sections are located in the capital cities of Western Australia, South Australia, Victoria, New South Wales, and Queensland respectively, the distances between

them being 1500, 500, 600, and 600 miles—about 3200 miles between the two extremes. Regular monthly meetings are held in each section, and each President usually finds it possible to visit each section at least once during his term of office. Council meetings are held twice each year, the venue alternating between Victoria and New South Wales. The Australian Society cannot at present afford to publish its own journal, but is fortunate in having an allocation of about fourteen pages in the *Textile Journal of Australia*, of which every member receives a copy each month.

The Australian Society keenly appreciates its affiliation with the Society of Dyers and Colourists in Great Britain, which is affectionately referred to in Australia as the "parent Society". The recent affiliation of the Dyers and Colourists Association of South Africa is another example of the progress of the Society. This was a convenient opportunity of suggesting that a bond much closer than that of affiliation with the kindred societies might be given some consideration.

Finally, on behalf of the visitors present, Mr. Castle thanked the President and members of the Society for their hearty welcome, for the privilege of being with them that evening, and for their fine hospitality.

PRESENTATION OF DIPLOMAS OF HONORARY MEMBERSHIP TO MR. F. SCHOLEFIELD AND DR. C. M. WHITTAKER

Mr. FRED SMITH (*President of the Society*) presented Diplomas of Honorary Membership to Fred Scholefield, M.Sc., F.R.I.C., F.T.I., F.S.D.C., and Croyden Meredith Whittaker, D.Sc., F.T.I., F.S.D.C., both awarded "in recognition of sustained and outstanding services to the Society". Mr. Smith said that Mr. Scholefield and Dr. Whittaker had probably done more than anyone previously to apply academic knowledge to the practice of dyeing and printing.

PRESENTATION OF THE SOCIETY'S SILVER MEDAL TO MR. C. C. WILCOCK

The President presented the Silver Medal of the Society to Clifford Collier Wilcock, A.R.T.C. (Salford), F.T.I., F.S.D.C., the award having been made "for exceptional services to the Society and to the tinctorial and allied industries". Mr. Smith referred to Mr. Wilcock's continued hard work for the Society, especially in the Manchester and London Sections.

AWARD OF OTHER MEDALS

The President read a letter from George Stuart James White, M.A., F.T.I., F.S.D.C., written from Harvard, U.S.A., in which he expressed his great regret at being unable to be present that evening, and also his deep appreciation of the honour of being awarded the Society's Gold Medal "for exceptional services to the Society and to the tinctorial and allied industries".

The Research Medal of the Worshipful Company of Dyers for 1953–1954 had been awarded to Ramsay John Hannay, B.Sc., F.R.I.C., F.T.I., F.S.D.C.

COMMUNICATIONS

The Influence of the Physical State of Dyes upon their Light Fastness*

G. BAXTER, C. H. GILES, Miss M. N. MCKEE, and N. MACAULAY

From a study of the quantitative relationship between light fastness and dye concentration conclusions are drawn regarding the physical state of dyes in air-dry substrates, on the basis of a simple theoretical model representing the nature of particle growth with increase in concentration.

Only one type of dye–substrate system, viz. a water-insoluble *meroc*yanine dye in solid solution in collodion, has been found to obey the normal photochemical laws for ideal systems. All other systems, whether involving water-insoluble or water-soluble dyes, behave non-ideally: i.e. the dye appears to exist in the form of aggregates or crystals (usually submicroscopic) and to obey a fading law in which the rate-affecting factor is the extent of the dye–air interface. Treatments which cause microscopically visible growth of such particles, e.g. soaping of azoic-dyed cellulose, raise the light fastness as anticipated.

Also in accordance with prediction, the light fastness even of water-soluble dyes is raised by increasing the porosity of the substrate; this is believed to result from the formation of larger aggregates of dye in the dry substrate. Thus the light fastness of direct cotton dyes is higher on highly porous ("gel") Cellophane film than on normal film, and on normal viscose rayon fibre than on highly oriented viscose rayon fibre; and acid wool dyes have higher fastness on the sulphuric-acid anodic film on aluminium than on the less porous chromic-acid film. A number of other examples of changes in light fastness, believed to be due to similar physical factors, are discussed.

The following two observations are reported— (*a*) undried "gel" Cellophane film slowly reverts to the normal form on keeping, even when wet; and (*b*) there is a linear relationship between the logarithm of percentage reflectance and the logarithm of total dye content for direct dyes on various viscose rayon fabrics.

Evidence was given in a previous paper[6] that the variation of light fastness which a dye may display when applied to different types of substrate must be due to the influence of both chemical and physical factors; and the nature of the chemical factors was discussed. The present paper discusses the physical factors influencing light fastness, and describes an investigation designed to demonstrate the specific effects of some of them.

The General Photochemical Law

It is well known that the reaction order of photochemical change, which is unity when only a

* A lecture describing part of this investigation was given (by C.H.G.) before the Scottish Section of the Society at the St. Enoch Hotel, Glasgow, on Tuesday, 23rd March 1954, Mr. R. D. Alexander in the chair.

small proportion of the incident active radiation is absorbed, falls steadily with increasing absorption, becoming zero when the whole of the incident active radiation is absorbed. This general law, however, has been derived largely from studies of "ideal" systems, i.e. gases or liquids in free kinetic motion, and "real" systems of solids, e.g. dyes present in fibres, may not necessarily conform with it, owing to complications arising from restricted access of radiation and/or of gaseous or liquid reactants. The present work was designed to examine a variety of such real systems and to determine the nature of the photochemical laws to which they conform, by a study of the quantitative relationship between fading rate and concentration of dye in substrate.

To enable the results to be most simply demonstrated, they are plotted as curves which will be referred to as *characteristic fading-order* or "CFO" *curves*. In these (cf. Fig. 2) the ordinate is the logarithm of the time t_F in which a given small percentage (10% in the present paper) of the initial quantity of dye is lost by fading; and the abscissa is the logarithm of the initial concentration C_0 of dye in the substrate.

A little consideration will show that the CFO curve for a dye behaving as an ideal photochemical system will have a slope of zero at low concentrations, steadily increasing with concentration up to some maximum value (calculated to be about 1·4—see Appendix, p. 234). In practice, however, only one type of dye–substrate system, out of a wide variety examined, has been found actually to behave in this way. This is a system of certain solvent-soluble dyes in collodion film (Fig. 5). Here the dye and the collodion are first mixed in acetone solution, and the films are formed by rapid evaporation of the solvent. It seems highly probable that this treatment leads to the formation of a "solid solution" of dye in collodion, i.e. a true molecular dispersion of dye randomly distributed amongst the molecular chains of the polymer. Such a system would seem to conform closely with ideal gaseous or liquid systems, as indeed is confirmed by the shape of the CFO curve.

All the other systems examined, however, give linear CFO curves with positive slope. It is believed that this is best explained by assuming that the dye is distributed in the substrate as discrete particles or aggregates, according to certain simple laws, which are now discussed with reference to a theoretical model intended to represent all dye–fibre systems, whether the dye is soluble in water or not.

Theoretical Model of the Light Fading of a Dye deposited on a Solid Substrate

There is evidence that the light fading of a dye on a fibre depends on the presence of atmospheric oxygen and water vapour[*]. The fading reaction must, therefore, occur at or near the dye–air interface. It will be assumed here that it occurs at the molecules in the surface layer of dye; and that the rate-affecting factor is the extent of this interface, i.e. the surface/weight ratio of the dye.

The model consists of a dye spread in two dimensions as a series of discrete particles (see Fig. 1) freely exposed to air and water vapour and evenly irradiated (by either directional or diffuse illumination).

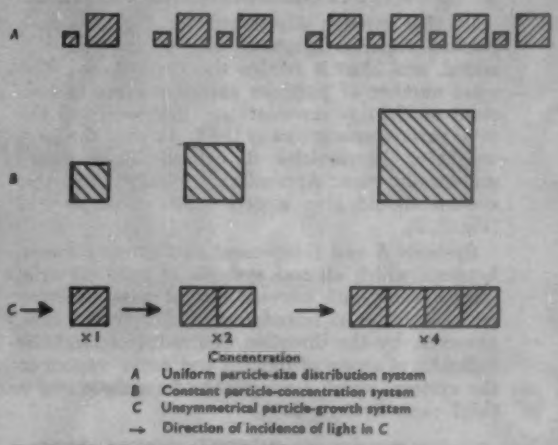
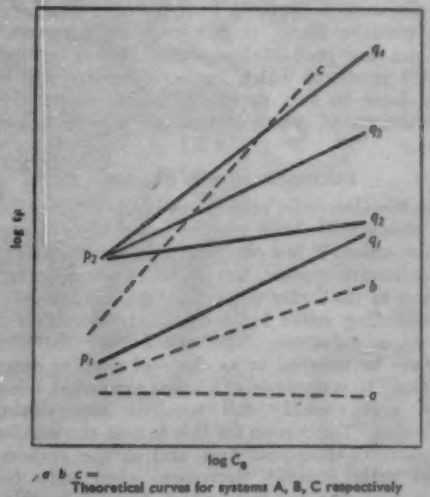


FIG. 1— Ideal Systems of Growth of Dye Particles with Increase in Total Concentration, in Ratio 1 : 2 : 4

The manner in which the fading rate of the dye in the theoretical model changes with increase in concentration will now be discussed with respect to three types of idealised systems.

A. UNIFORM PARTICLE-SIZE DISTRIBUTION SYSTEM

This system, illustrated by Fig. 1A, may consist of particles of any number of sizes or shapes provided that at each concentration of dye the proportion of particles of each particular size or shape remains the same. Thus the total number of



a b c — Theoretical curves for systems A, B, C respectively

P_2q_2, P_2q_3, P_2q_4, P_1q_1, P_1q_2, P_1q_3 — Typical curves obtained in practice

FIG. 2— Typical Characteristic Fading-order (CFO) Curves

[*] Most of the published evidence concerns cellulosic fibres[6]. Duff[9] has, however, shown that logwood, dyed on mordanted wool, does not fade in absence of atmospheric oxygen.

particles and the surface area increase in direct ratio to the total weight, so that the surface area/weight ratio is constant, and hence the time taken for a given proportion of dye to fade is also constant. The fading reaction is thus of the first order and the CFO curve is a horizontal line (Fig. 2, *a*).

B. CONSTANT PARTICLE-CONCENTRATION SYSTEM

In this system, illustrated by Fig. 1B, each particle expands symmetrically as more dye is added, and thus it retains the same shape. The total number of particles therefore remains constant as the dye concentration increases, but the total surface area grows as the $\frac{2}{3}$ power of the total weight of the particles. It can readily be shown graphically (see Appendix, p. 234) that this system should give a CFO curve of slope 0·12 (Fig. 2, *b*).

Systems A and B represent two extreme forms, between which all real systems of solid material must develop with increase in total mass. Another variable factor is introduced in the present case, however, by the direction of irradiation and the difficulty of access of oxygen and water vapour to the system, which leads to the formulation of a third case, System C.

C. UNSYMMETRICAL PARTICLE-GROWTH SYSTEM

This system is illustrated by Fig. 1C. With increase in total dye concentration, each particle grows, but in only one direction. In the extreme case, where the illumination and the oxygen and water vapour reach only the side of the particle which does not increase in size, the total surface area of dye being irradiated remains the same at all dye concentrations, and hence the total *amount* of dye faded is constant. The fading reaction is then of zero order, and by a graphical method (see Appendix, p. 234) it can be shown that the CFO curve has a slope of 1·4 (Fig. 2, *c*).

If a system is found to have a linear CFO curve with positive slope, it follows that aggregated particles are probably present. This is an important point, to which further reference will be made later in this paper. (Possible alternative explanations of positive slope are also discussed below.)

FADING REACTION ORDERS

The reaction order referred to in the descriptions of Systems A–C above may be called the *apparent reaction order*. It is a characteristic parameter of a dye–substrate system, but it bears no necessary relation to the order determined by studying the rate-of-fading curve for a single concentration of dye on a substrate. The latter may for convenience be referred to as the *real reaction order*; it is likely to commence at a value somewhat below unity, and steadily fall towards zero during irradiation. The reason for this is that the smaller dye particles disappear first, and so the surface : weight ratio steadily decreases, until only the largest particles are left, which have a reaction order approaching zero. Yet a third order may be defined, the *ideal reaction order*, which is that of the dye when fading in a true molecular dispersion under uniform irradiation.

The three reaction orders thus defined have the same numerical value, i.e. unity, in only one special case, where the dye is spread as a true monolayer in the substrate and only a small proportion of the active incident radiation is absorbed. The real reaction order then remains constant throughout irradiation, because no change in average particle size can take place.

EFFECT OF POROSITY OF THE SUBSTRATE

In the ideal systems just described it is assumed that the dye is extended in two dimensions only. In real systems the dye will be distributed in three dimensions throughout the submicroscopic pores of the substrate, and the rate-determining factor in fading might then be, not the surface area of the dye, but the pore size of the substrate, since this would control the rate of diffusion of air and water to the surface of the dye. The experimental evidence given later, however, is not in favour of the pore size being the rate-controlling factor, and in the subsequent argument it is assumed not to be so.

EFFECT OF CHANGES IN AGGREGATION UPON THE CHARACTERISTIC FADING ORDER CURVE

We may now consider the probable effects upon light fastness of different forms of dye crystal growth or aggregation in real dyed systems.

Consider the CFO curve of a dye–fibre system which on fading gives a curve p_1q_1 (Fig. 2), and suppose that some treatment is applied which causes no chemical change but which promotes physical aggregation of dye particles. This aggregation reduces the surface/weight ratio of the dye and so raises the light fastness at all concentrations. The CFO curve of the system is therefore raised, but its slope may decrease, increase, or remain constant, giving, say, curves p_2q_2, p_3q_3, p_4q_4 respectively. The reasons for these changes may be stated as follows—

(*i*) *Decrease of Slope*

The dye particles in the upper concentration ranges are already approaching a size limit determined by their own crystal habit or by restraints imposed by the structure of the substrate, and thus the higher the concentration the fewer the number of particles there are which grow.

(*ii*) *Increase of Slope*

The dye becomes aggregated more readily the closer its particles are to each other, i.e. the higher its concentration.

(*iii*) *Constancy of Slope*

This is obviously an intermediate stage between conditions (i) and (ii), and would mean that the dye shows a uniform tendency to aggregate whatever the concentration; i.e., however many particles are originally present, each group of a given number of them will coalesce into a single unit.

The reverse effects on the CFO curves will naturally follow the application of any treatment which causes purely physical disaggregation.

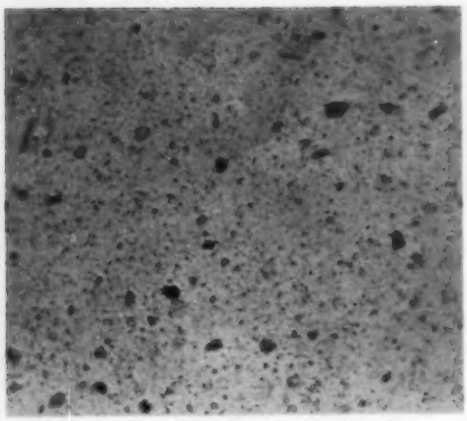


FIG. 3A — Dispersion of *mero*Cyanine Dye in Gelatin (I) (× 500)

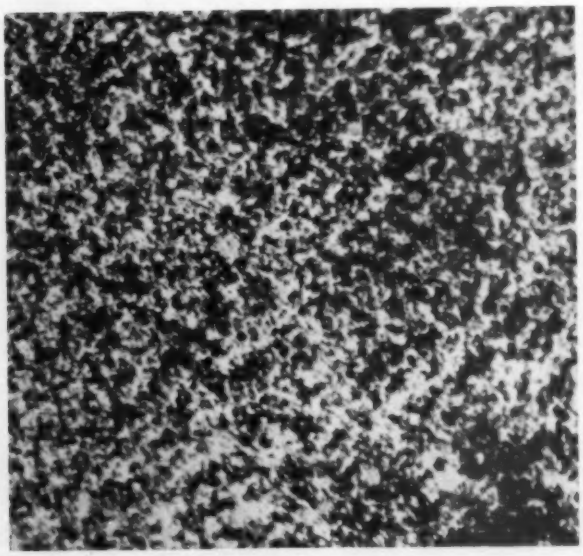


FIG. 3B — Dispersion of Azo Dye (*p*-Anisidine→2-Naphthol) in Methylethylcellulose (× 500)

FIG. 6A — Azoic-dyed Cellophane before Soaping (× 500)

FIG. 6B — Azoic-dyed Cellophane after Soaping (× 500)

A corollary of these arguments is that, in the absence of restraints imposed by substrate structure, an increase in aggregation, i.e. an increase in the maximum size of particle present, should always cause a rise in the slope of the CFO curve, and a decrease in aggregation should cause a fall. The explanation is that, as the maximum size of aggregate increases, assuming a chance distribution of particle sizes, the number of possible smaller sizes which may be present rises, i.e. the particle-size distribution becomes less uniform. But the most uniform particle-size system (System A above) has a CFO curve of lowest possible slope (zero), and a decrease in uniformity thus causes a rise in slope.

The case of a *negative* slope for the CFO curve is not considered, because this condition has not so far been encountered in any system examined. If found, it would of course mean that the dye aggregates more at low than at high concentrations.

The following discussion shows, by reference to earlier work and to the present investigation, how the above theoretical predictions are borne out in practice.

Previously Published Work

Perhaps the most definite experimental evidence hitherto obtained connecting light fastness with the physical state of the dye in the fibre is that of Bean and Rowe [3], who made a careful study of the effects of a number of aftertreatments upon azoic dyes on cellulosic fibres. They observed that treatments, e.g. soap-boiling or steaming, which caused crystal growth of such dyes, sufficiently pronounced to be visible under the microscope, increased the resistance to fading. In their own words—

> The fastness to light of material dyed with a given insoluble azo colour increases with increase in the size of the pigment particles remaining in the fibre. . . . the increased fastness to light . . . appears to us to be due merely to the decrease in the surface area of the colouring matter exposed, although it is also possible that crystalline aggregates may be more resistant to light than amorphous ones. Comparative exposures of these colouring matters in substance in varying degrees of subdivision also showed that coarse particles are faster to light than finer particles of the same compound.

No quantitative data are given*.

Very little other information appears to have been published which demonstrates any definite connection between particle size and fastness. Such a connection is, however, generally believed to be shown by the behaviour of vat dyes on nylon, on which they generally have much lower light fastness than on cellulosic fibres. The difference has been attributed to their smaller particle size on nylon [8], and processes have been described for improving the fastness by treatments which appear to involve swelling of the fibre, which would enable the dye particles to increase in size. Thus, the dyed material may be steamed [11]; it may be treated in a boiling aqueous solution of a polyhydroxy compound, e.g. ethylene glycol [10], or in solutions of aromatic compounds containing hydroxyl and/or carboxylic acid groups [5]; or salicylic acid may be added to the developing liquor for water-solubilised vat dyes of the Indigosol type [26].

* Light fastness reference standards had not then been established in this country.

I—Direct Tests of the Theoretical Model Systems

Demonstrations of the validity of the model systems were made in the present work with certain water-insoluble dyes as follows—(1) by fading a dye after introduction into films in markedly different states of dispersion, and (2) by fading azoic dyes in cellulose before and after aggregation by soaping.

1. FADING OF DISPERSIONS, ETC. OF *MEROCYANINE* AND AZO DYES

For this test two *mero*cyanine dyes and one azo dye were used. Two or more of the following methods of preparation of films were employed in each case—

(i) Hand grinding to relatively coarse particles, followed by dispersion in films of gelatin or methyl ethyl cellulose (cf. Fig. 3) (System A).

(ii) Dissolution in acetone followed by precipitation in an aqueous solution of gelatin or methyl ethyl cellulose, with subsequent film formation.

This should give a series of films also conforming with System A, provided that the dye is quite insoluble in water, but the average particle size should be less than that of the series (i) films.

(iii) Dissolution in collodion followed by film formation.

In all the films of series (i) and (ii) particles were easily visible under the microscope (Fig. 3A and 3B), but none could be seen in the collodion films of series (iii).

In each case a series of different dye concentrations in the substrate were used, and the CFO curves were determined as described in the Experimental Section (pp. 233–234).

The results (Fig. 4) confirm the predictions of the theoretical model in two respects—(*a*) the System A dispersions give CFO curves with slopes nearly or quite zero; and (*b*) the rate of fading increases, i.e. the *y*-axis intercept of the CFO curve falls, with increase of surface/weight ratio of dye.

The slight positive slope of some of the series (i) and (ii) curves may be due either to the "screening" effect of closely packed particles discussed below, or more probably to some differential migration of particles during the preparation of the films. The azo dye collodion film curves have more pronounced slope, perhaps owing to some (submicroscopic) crystallisation of the dyes during the removal of the solvent in drying.

The results for the first *mero*cyanine dye and the azo dye* are shown in Fig. 4 and 5. Preparations by methods (i) and (ii) may be classed under model system A, because their particle-size distribution should be uniform at all concentrations. As would therefore be anticipated, the CFO curves in gelatin

* The scatter of points for the azo dye is due to the difficulty of obtaining even dispersion of the crystals in the dried film.

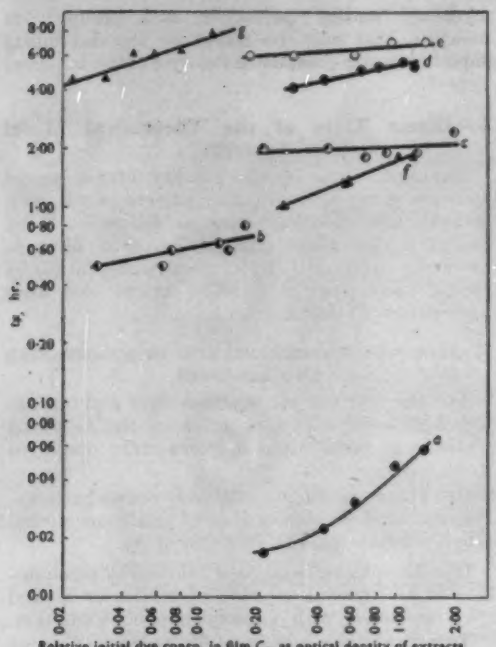


FIG. 4— CFO Curves for AZO and *mero*Cyanine Dyes in Various Films

have virtually a zero slope. Those in methyl ethyl cellulose have a slight positive slope. This is probably due to some differential migration of particles during the drying of the films. In accordance with prediction, the intercepts of the curves on the *y*-axis rise with increase in average particle size as judged visually under the microscope*.

The second *mero*cyanine dye proved to be too resistant to light to be faded in a reasonable period, except in the finest dispersion, prepared from the acetone solution. This gave a slope (curve not shown) of about 0·5; evidently the particle size distribution had not remained uniform over the range of concentrations under the precipitation conditions used, probably on account of the partial solubility of the dye in water.

In systems of this nature, the fastness measured by the present method is obviously largely determined by the rate of fading of the smaller particles, which are probably invisible in the illustration (Fig. 3A). The largest would fade at a much slower rate than that observed. It is noticed, for example, that fading is much more rapid in the earlier stages of the irradiation than later.

* In view of the wide range of particle sizes present in these dispersions it was not considered worthwhile to attempt to make any exact correlation between particle size and rate of fading. Some dispersions of vat dyes of known and more uniform particle size were examined, but their fading rates proved to be too low for tests to be practicable.

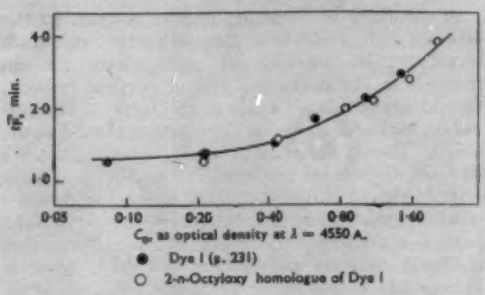


FIG. 5 — CFO Curves for *mero*Cyanine Dyes in Collodion Film

According to the classification of the previous paper [6], fading on gelatin is of a different nature from fading on either collodion or methyl ethyl cellulose; yet it is interesting to note that this does not affect the conformity of the curves to the theoretical model, which should, of course, be independent of the chemical nature of the fading reaction.

2. FADING OF AZOIC DYES, SOAPED AND UNSOAPED

Regenerated cellulose films (Cellophane) dyed with typical azoic combinations were faded in the unsoaped state and after soap-boiling. The soap-boiling was sufficient to cause the growth of particles in the film which were visible under the microscope; no particles were visible before this treatment (Fig. 6). Several tests were made (Fig. 7 and 8; cf. also Fig. 2[12]) and in each case the expected effect upon the CFO curve is actually observed: the intercept of the curve on the *y*-axis rises markedly on passing from unaggregated to aggregated dye. The fastness for any given depth of dyeing is in fact approximately doubled in this process. Even on allowing a film to soak in cold water overnight, a slight change in the CFO curve is observed (Fig. 8). The slopes of the curves change slightly after the treatments, either decreasing or increasing, no doubt because of changes in the nature of the aggregation processes, as already discussed.

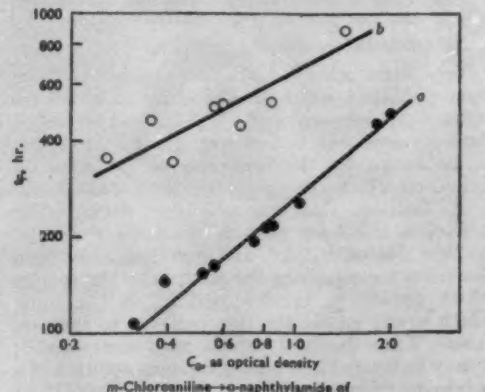
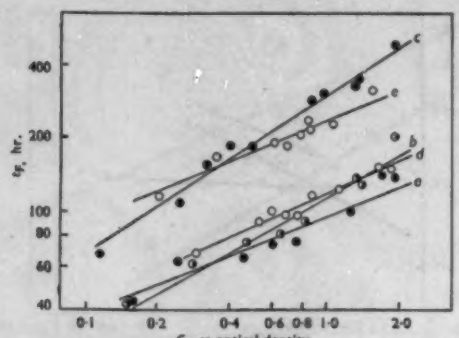


FIG. 7— CFO Curves for Azoic Dye on Cellophane



m-Chloroaniline→α-naphthylamide of 2:3-hydroxynaphthoic acid (Brentamine Fast Orange GC→Brenthol AN (ICI))
(a) Untreated (b) Steeped in water overnight (c) Soaped

m-Chloroaniline→anilide of 2:3-hydroxynaphthoic acid (Brentamine Fast Orange GC→Brenthol AS (ICI))
(d) Untreated (e) Soaped

FIG. 8— CFO Curves for Azoic Dyes on Cellophane

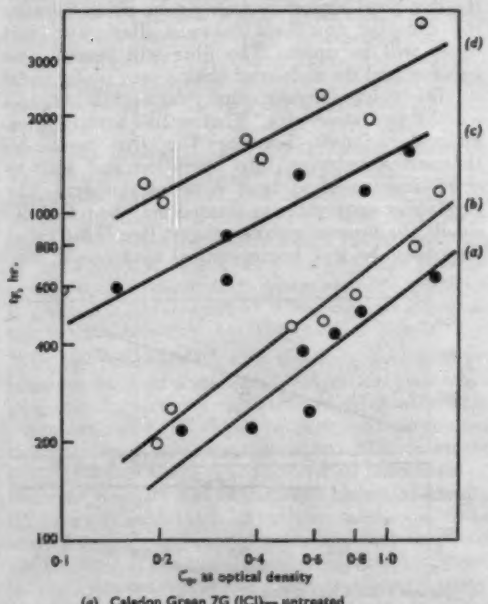
II—Indirect Tests of the Theoretical Model Systems

So little is known of the physical state of dyes in fibres that it is hardly possible to make a direct test of the soundness of the proposed theoretical model by other than the few procedures just described. In the remainder of the work only indirect tests were possible.

1. WATER-INSOLUBLE DYES

(i) *Anthraquinonoid Vat Dyes on Nylon*

Examination of nylon film dyed with two vat dyes before and after treatment with a swelling agent showed (Fig. 9) that the CFO curves are higher in the treated film, no doubt on account of the more ready growth of large particles when the fibre is swollen.



(a) Caledon Green 7G (ICI)— untreated
(b) Caledon Green 7G (ICI)— treated with cinnamic acid
(c) Caledon Yellow GN (ICI)—untreated
(d) Caledon Yellow GN (ICI)— treated with cinnamic acid

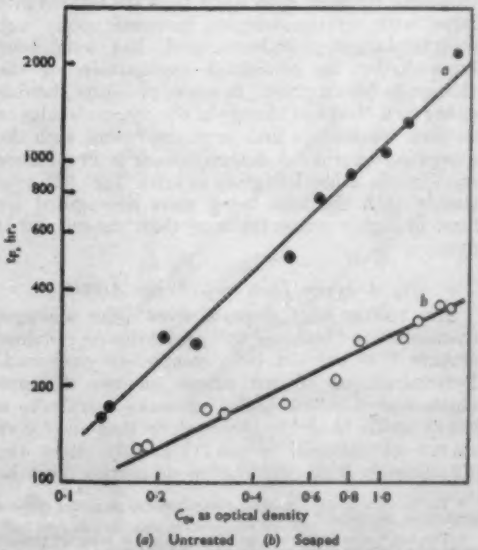
FIG. 9 — CFO Curves for Vat Dyes on Nylon Film

(ii) *Indigoid Vat Dyes on Cellulose*

Soaping aftertreatment is normally given to vat-dyed cellulose materials, in order to develop and stabilise the true colour. The effects of this treatment on cellulose film dyed with vat dyes have been examined by Sumner, Vickerstaff, and Waters [27] employing spectrophotometric methods with polarised light. They used undried films, and found that soaping appears to accelerate the normally slow change of leuco dye to its oxidised form. At the same time the molecules of leuco compound, initially oriented parallel to the cellulose molecular chains, take up a position at right angles to this direction; it is suggested that needle-shaped crystals of oxidised vat dye are formed in which the individual molecules are perpendicular to the longest axis. In an assessment of the light fastness of dyeings on cotton, it was found that one was faster after soaping, one less fast, and the remaining eight showed differences of a minor order, probably within experimental error.

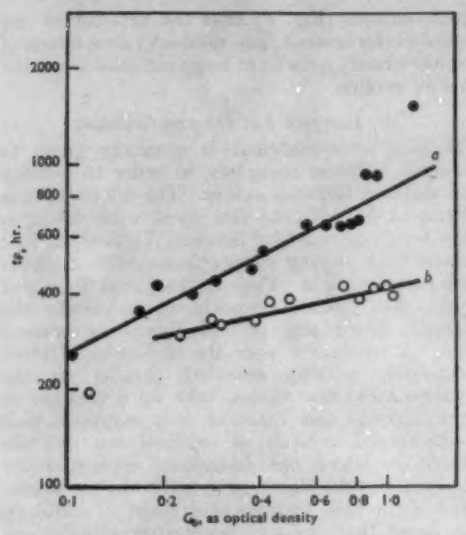
Sumner, Vickerstaff, and Waters [27] show electron micrographs of a vat dye dispersion before and after an ageing process rather more severe than that given in the normal soaping (no crystals could be seen in the electron micrographs of dyed and soaped films). These show that the process changes the form of the dye from rather compact amorphous lumps to fine filamentous crystals, and an inspection of the micrographs suggests that in this case the ageing process does increase rather than decrease the surface/weight ratio.

In the present investigation, two indigoid vat dyes were applied to Cellophane film, and the



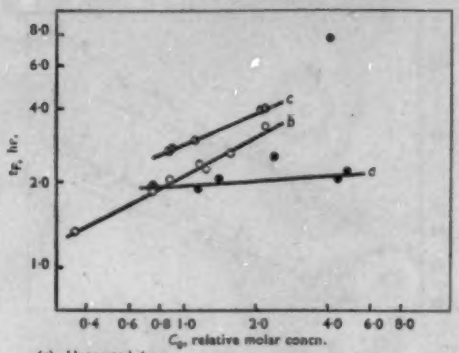
(a) Untreated (b) Soaped

FIG. 10— CFO Curves for Durindone Blue 4BC (ICI) on Cellophane Fibre



(a) Untreated (b) Soaped

FIG. 11— CFO Curves of Durindone Red 3B (ICI) on Cellophane Film



(a) Untreated dye
(b) Molybdophosphoric acid lake (equimolar)
(c) Molybdophosphoric acid lake (25% molar excess of acid)

FIG. 12— CFO Curves of Victoria Blue BO in Lithographic Varnish Film

dyeings were examined both untreated and after soaping. It was necessary to choose indigoid dyes, of relatively low fastness, because of the exceedingly lengthy exposures which would have been required with vat dyes of normal high fastness on cellulose. Even so, some of the fading tests had to be carried on for well over 1000 hr. The CFO curves (Fig. 10 and 11) appear to demonstrate an increase in surface/weight ratio after soaping, apparently in agreement with the results of Sumner *et al.*[27].

(iii) *Lakes of Basic Dyes with Complex Acids*

The considerably improved light fastness exhibited by basic dyes when they are formed into lakes with certain complex inorganic acids, e.g. molybdotungstophosphoric acid, has long been known*, but no conclusive explanation of the change has been given. It must, of course, be due either to a chemical change in the dye molecules or to their coalescence into large aggregates with the inorganic reagent. A determination of CFO curves on Victoria Blue BO gives results (Fig. 12) consistent with the lake being more aggregated (at least in higher concentrations) than the untreated dye.

(iv) *Disperse Dyes on Cellulose Acetate*†

The reason why disperse dyes differ amongst themselves in "building up" properties on cellulose acetate [cf. 29] has not been completely explained. Determinations of CFO curves on two disperse blues, one of which builds up more readily to a heavy shade than the other, show that they give curves of different slopes (Fig. 13); thus the difference in their building-up properties must be due to differences in their state of aggregation in the air-dry fibre.

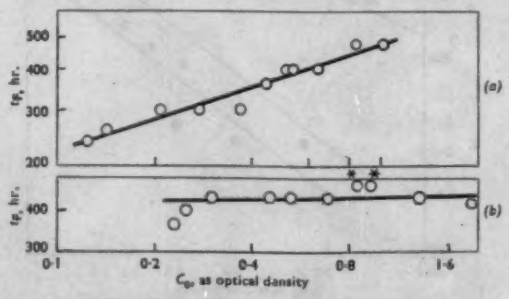
* For an account of the history, preparation, and properties of these compounds, see Miller[19].

† This test was made to examine the possibility of using the present method for studying properties other than light fastness.

2. WATER-SOLUBLE DYES

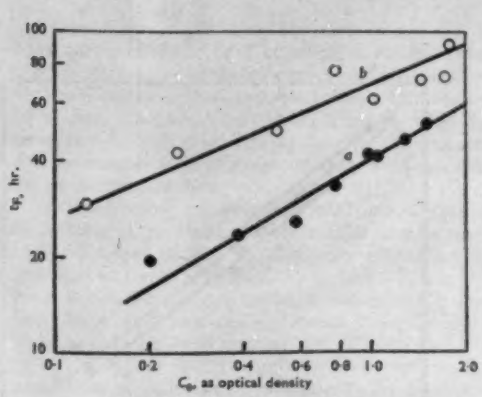
The next experiments were designed to determine whether the application of the theoretical model could be extended to water-soluble dyes.

Very little is known of the physical state of such dyes in air-dry fibres. Current beliefs [cf. 28] favour a mechanism in which each molecule of adsorbed dye is individually held during the dyeing process by part of the molecular chains of a fibre. This mechanism would produce a monodisperse system or a monolayer (System A). When the bulk of the water is removed, however, changes in the state of the dye must almost certainly take place, because the complex equilibria between fibre, dye, and water will be upset. The fibre will become less swollen, and its molecular chains may under these conditions have higher affinity for each other than for the dye molecules. This would permit intermolecular forces between the dye molecules themselves to come into operation and lead to crystallisation of at least some of the dye. The crystals or aggregates so formed may be relatively small: the present results suggest (see below) that they may be well below optical microscopic size.



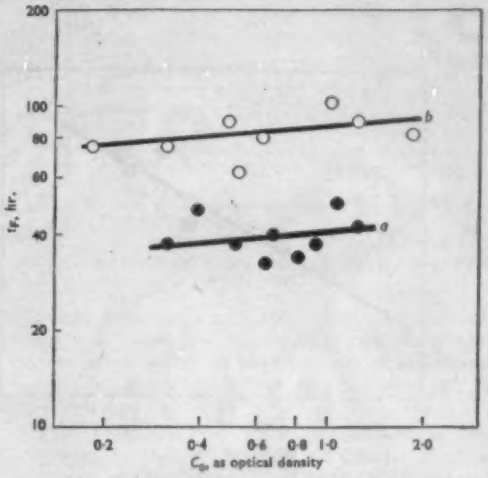
(a) Duranol Brilliant Blue G (ICI)
(b) Duranol Brilliant Blue 8N (ICI)—the two points marked (*) were calculated by back-extrapolation of the later stages of the fading curves, the earlier stages being irregular

FIG. 13 — CFO Curves for Disperse Dyes on Cellulose Acetate Film



(a) Normal film (b) Gel film, fresh

FIG. 14— CFO Curves for Chlorazol Sky Blue FF (ICI) on Cellophane Film of Different Porosities



(a) Normal film (b) Gel film, fresh

FIG. 15— CFO Curves for Chlorazol Rose B (ICI) on Cellophane Film of Different Porosities

The only observations reported upon this subject, so far as we are aware, concern various dyes on cellulose and a few acid dyes on wool. Thus Preston [23] and Morton [20] have recorded the behaviour of (air-dry) dyed cellulose fibres observed under the microscope, in polarised light. By comparison of simultaneous observations on solid dyes and streaming dye solutions, Morton concluded that direct dye molecules are attached lengthwise, i.e. with their long axes parallel to the cellulose molecular chains, and that in the solid powder or crystals they are arranged with their long axes across the length of the crystal.

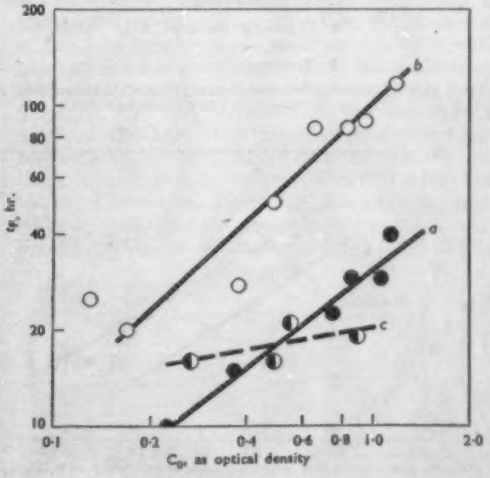
A few observations of the state of dyes in air-dry wool fibre were made by Astbury and Dawson [1], who studied, by X-ray diffraction, the setting properties of samples of wool dyed with a few acid dyes. They detected free crystallites of two dyes— Solway Blue B (*Colour Index* No. 1054) and Solway Blue SEN (*C.I.* 1053)— but no crystallites were detected in fibres dyed with either Crystal Ponceau (*C.I.* 89), Coomassie Navy Blue 2RN (*C.I.* 289), or Orange II (*C.I.* 151); presumably any amorphous dye aggregates present would not be detectable. Speakman and Smith [25] also noticed the presence of free particles of solid dye, large enough to be readily visible under the microscope, in wool dyed at 60·5°C. with Solway Blue SEN in presence of sulphonated castor oil. No particles appeared in fibres dyed at 97°C. Similar observations were reported by Royer and Maresh [24].

In the present work two methods were used to discover whether water-soluble dyes are present as aggregated particles in air-dry substrates— (*a*) examination of the slopes of their CFO curves, on the principles already stated (p. 219); and (*b*) comparison of their light fastness on substrates of different porosity but of the same chemical nature. Three such substrates have been used, viz. films and fibres of regenerated cellulose, and anodised aluminium. If the dyes aggregate, then an increase in pore size may promote the formation of larger particles with a consequent increase in light fastness; on the other hand, if only monolayers of dye are present, increased porosity would cause no increase in light fastness for a given concentration of dye.

(i) *Direct Cotton Dyes applied to Regenerated Cellulose Films of Different Porosity*

Two types of Cellophane regenerated cellulose film were used— normal dried film and gel film, i.e. material of higher porosity*, which had not been dried after coagulation. In accordance with prediction, the light fastness of two typical direct cotton dyes— Chlorazol Sky Blue FF (ICI) (*C.I.* 518) and Chlorazol Rose B (ICI)— is considerably higher at all concentrations on the gel film than on the normal dry material (Fig. 14 and 15). Also, all the curves have positive slope.

* The respective pore sizes were stated to be of the order of 20 A. and 30 A.[3].



(a) Normal film
(b) Gel film, fresh
(c) Gel film, aged

FIG. 16— CFO Curves for Chlorazol Sky Blue FF (ICI) on Cellophane Film of Different Porosities

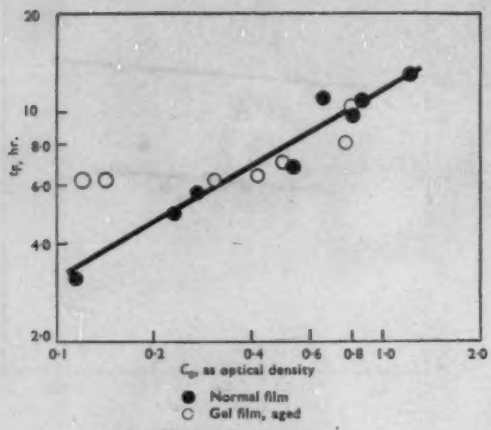


FIG. 17 — C_{F0} Curves for Chlorazol Orange PG (ICI) on Cellophane Film

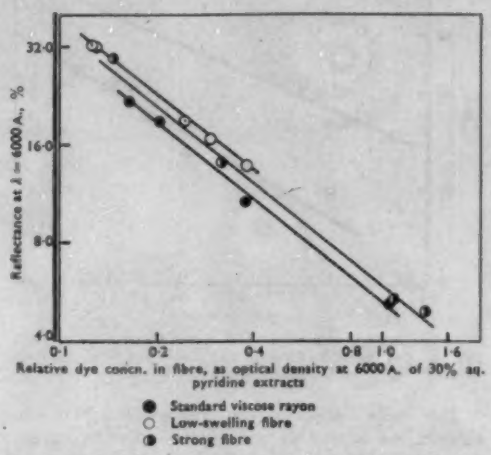


FIG. 20 — Relationship between Concentration of Chlorazol Sky Blue FF and Reflectance of Viscose Rayon Fabrics (Series I)

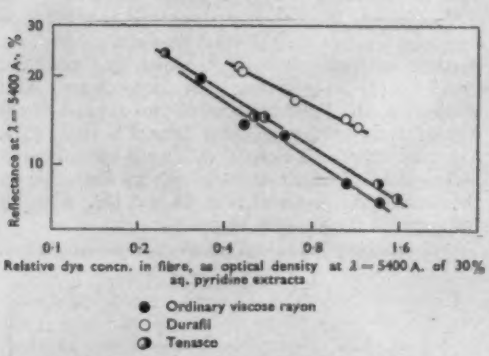


FIG. 18 — Relationship between Concentration of Chlorazol Rose B (ICI) and Reflectance of Viscose Rayon Fabrics

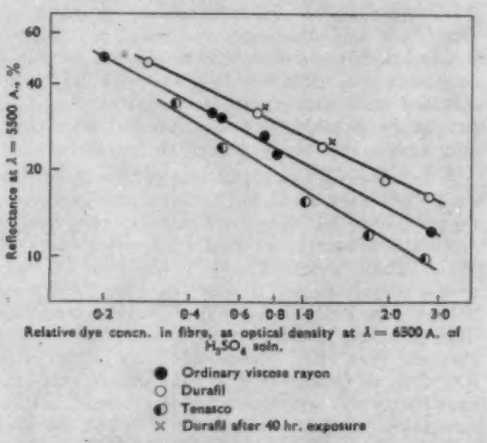


FIG. 21 — Relationship between Concentration of Chlorazol Fast Helio 2RK (ICI) and Reflectance of Viscose Rayon Fabrics

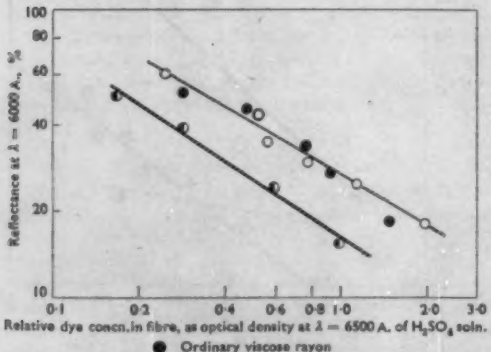


FIG. 19 — Relationship between Concentration of Chlorazol Sky Blue FF and Reflectance of Viscose Rayon Fabrics

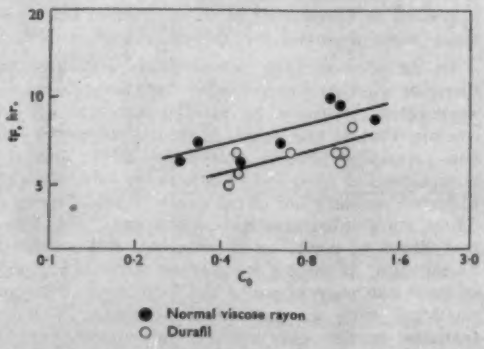


FIG. 22 — C_{F0} Curves for Chlorazol Rose B on Viscose Rayon Fabrics

These facts imply that the dye is present at least partly in the form of aggregates, and also that the maximum size of these aggregates is determined by the porosity of the substrate. The gel film must, of course, contract when dried after dyeing, but nevertheless this does not appear to prevent the dye aggregates in it from growing larger than they do in the normal film.

An interesting observation was made during these tests: it was found that this difference between the two types of film applies only when the gel material is fresh. Dyeings on material which had been stored for several weeks, even though it had been kept wet, had no significantly higher light fastness than those on normal film (Fig. 16 and 17). It would appear that the gel slowly reverts to the normal state on keeping, presumably by a process of slow crystallisation of the cellulose.

Some other evidence is available that this process occurs [4].

(ii) *Direct Cotton Dyes on Viscose Rayon of Different Degrees of Crystallinity*

The CFO curves were obtained for direct cotton dyes applied to two series of viscose rayon staple-fibre fabrics similar in construction but prepared from filaments of different degrees of crystallinity.

First the relationship between the actual dye content of the fibres and their reflectance was determined by using the spectrophotometer with reflectance attachment. It is interesting to note that there is actually a linear relationship between the logarithm of the dye content and the logarithm of the reflectance (Fig. 18–21). The interdependence of these two factors is therefore, at least in the present circumstances, much simpler than might be expected from the rather complex formulae which have sometimes been proposed on theoretical grounds.

It follows from this relationship that any given percentage gain of reflectance will represent a constant percentage loss of dye over the whole concentration range used, and in plotting Fig. 22–25 the reflectance gains corresponding in each case with 10% loss of dye content have been used in determining t_F values. Thus these curves are all comparable with those for transparent films. It was found (see Fig. 21) that the relationship

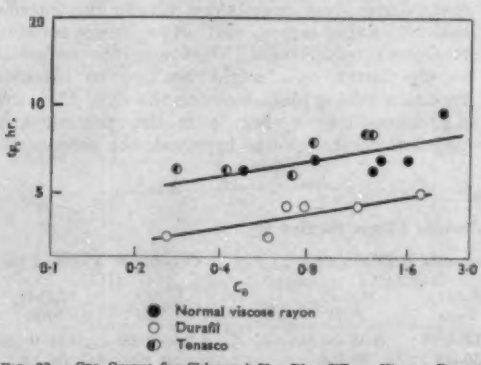


FIG. 23 — CFO Curves for Chlorazol Sky Blue FF on Viscose Rayon Fabrics (Series II)

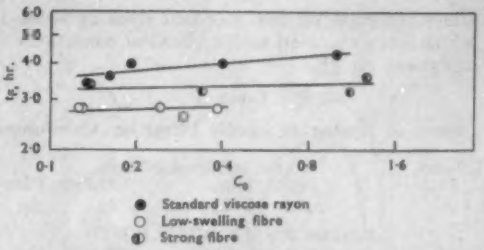


FIG. 24 — CFO Curves for Chlorazol Sky Blue FF on Viscose Rayon Fabrics (Series I)

between reflectance and dye content altered only very little when two fabrics were irradiated until considerably faded, so that the use of reflectance values in studying fading is considered to be justified.

The results (Fig. 22–25) demonstrate the anticipated higher fastness of each dye on ordinary viscose rayon than that of the corresponding dyeings on the more crystalline Durafil fibre.

In one case Tenasco-type fibre gave an intermediate fastness (Fig. 24) and in another (Fig. 23) it resembled ordinary viscose rayon.

In three of these sets of tests the dyeings of lower fastness are obtained on fibres (of Durafil) with a much finer filament than those of normal viscose rayon giving higher fastness (see Experimental Section, p. 231). This cannot, however, be attributed to any effect of the consequent differences in external surface areas of the fibres, because the fastness differences persist even when all the fibre types have almost the same filament size (Fig. 24; see also Experimental Section, p. 231).

(iii) *Acid Wool Dyes on the Anodic Film on Aluminium*

Films which readily dye with acid wool or mordant dyes may be prepared on the surface of aluminium by electrolysing it (as anode) in solutions of chromic or sulphuric acid. These films are almost the same chemically, consisting of γ-alumina (Al_2O_3). The film prepared in chromic acid is substantially pure; that prepared in sulphuric acid contains up to 13% aluminium sulphate [23], but there is no reason to suppose that this will affect the present results. Physically, however, the films differ in that the sulphuric acid film is much more porous. This is demonstrated by the dyeing

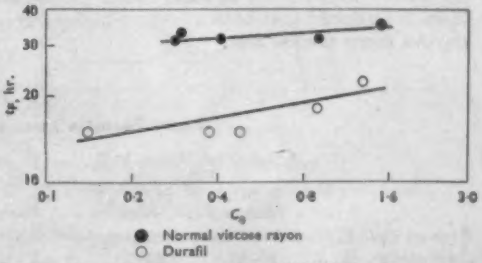


FIG. 25 — CFO Curves for Chlorazol Fast Helio 2RK on Viscose Rayon Fabrics

times recorded for two mordant dyes in Table I, which were obtained under identical conditions of agitation, etc.[15].

TABLE I

Rates of Dyeing of Anodic Films on Aluminium

Temp. (°c.)	Film and Dyeing Times			
	H_2SO_4 Film		H_2CrO_4 Film	
	$t_{0.5}$	t_{eq}	$t_{0.5}$	t_{eq}
	ALIZARIN RED IP (ICI) (*C.I.* 1027)			
40	5·0	1·60	34·0	60·0
60	2·6	0·60	11·0	30·0
75	1·2	0·25	—	—
80	—	—	9·0	16·7
	SOLWAY BLUE BNS (ICI) (*C.I.* 1054)			
20	—	—	16·0	100·0
40	0·90	0·90	12·0	53·5
60	0·50	0·60	8·0	30·0
75	0·25	0·25	—	—

$t_{0.5}$ = Time (hr.) of half-dyeing.

t_{eq} = Time (hr.) to reach equivalent degree of adsorption—0·2 mole/kg. for Alizarin Red IP and 0·08 mole/kg. for Solway Blue BNS.

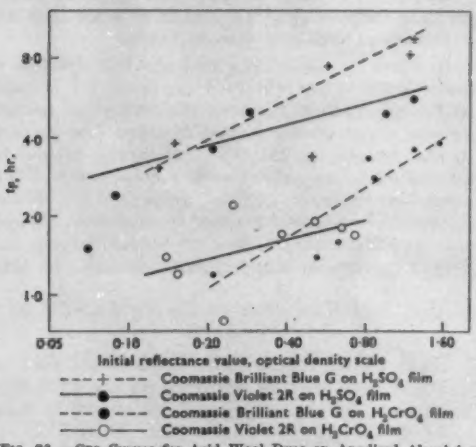


FIG. 26 — CFO CURVES for Acid Wool Dyes on Anodised Aluminium Films (Series I)*

*The data in Table III refer to the combined dye contents of both faces of the respective samples; the results plotted in Fig. 26 are calculated from separate exposures of the individual faces.

Two acid dyes were applied separately to both types of film and the CFO curves determined (Fig. 26). Experimental difficulties account for the rather wide scatter of some of the points, but there is no doubt that both dyes give higher curves on the more porous film.

The experiments were then all repeated, the sulphuric acid film being prepared this time on metal which had been first anodised in chromic acid solution, and then stripped, in order to give a matt background to facilitate reflectance readings. The dyeings were made at 50–60°c. for 30 min. with mechanical agitation, and an additional dye was included. Unfortunately, the results were less satisfactory than those of the first series, the fading-rate curves being particularly irregular, and it has not been practicable to plot them; nevertheless the mean data (summarised in Table II) confirm the earlier results.

The relationship established in this work between light fastness on the two types of film apparently holds for most dyes. In a list of seventeen commercial dyes in "full" shades[16] each has a higher fastness grading on sulphuric acid film than on chromic acid film, the mean fastness grades being about 7 and 5 respectively.

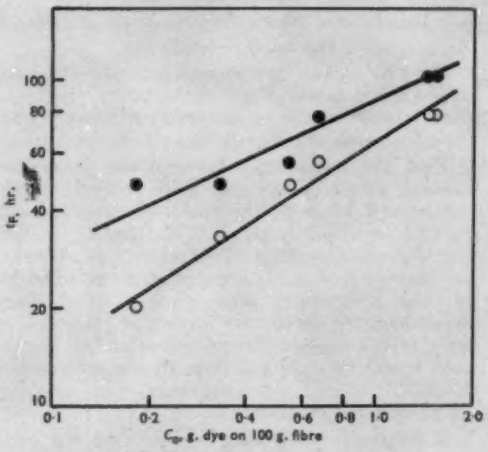


FIG. 27 — CFO CURVES for Durazol Red 2B (ICI) on Viscose Rayon (Visual Estimation)

(iv) *Examination of Possible Disaggregating Treatments*

An examination was made of two processes which are known to reduce the light fastness of many direct dyes on cellulose, viz. application of a cationic fixing agent and of a crease-resisting treatment respectively. Obviously the reduction in the latter case might be due to chemical reactions taking place between the dye, fibre, and e.g. formaldehyde, but it is also possible that physical effects may be involved: the pores of the

TABLE II

Light Fastness Data on Anodic Films (Series II)

	Acilan Fast Green 10 G (FBy)		Coomassie Brilliant Blue G 250 (ICI)		Coomassie Violet 2 RS (ICI)	
	H_2SO_4 Film	H_2CrO_4 Film	H_2SO_4 Film	H_2CrO_4 Film	H_2SO_4 Film	H_2CrO_4 Film
Dye on film, % ...	0·001–0·54	0·03–0·40	0·15–0·53	0·03–0·83	0·05–0·60	0·01–0·33
Reflectance, % ...	90–40	65–30	50–45	70–7·0	50–20	65–15
t_p (mean), hr. ...	3·0	1·1	6·0	2·0	1·5	1·0

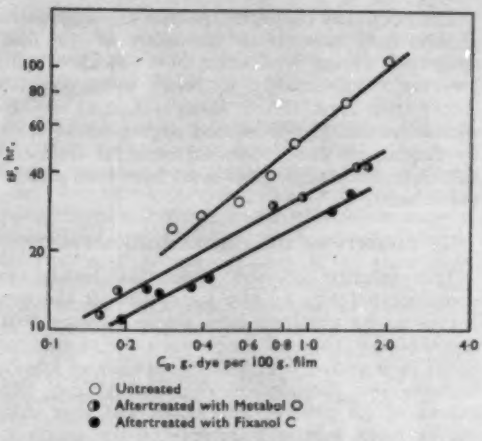


FIG. 28 — CFO Curves for Chlorazol Sky Blue FF on Cellophane Film

fibre may be blocked with resin, and so the size of any aggregates present may be reduced. The change in the CFO curve after the treatment (Fig. 27) is consistent with this.

In the case of the cationic fixing agents the possibility of any change in light fastness being due to chemical reaction between the agent and the dye seems remote. The only chemical reaction occurring must be the replacement of an inorganic cation (usually sodium) by the organic cation, and it seems unlikely that this can directly influence the photochemical reactivity of the dye anion. It is more likely to have an indirect influence by interference with the crystallisation of the dye, since the larger cation must disturb the crystal lattice structure. Some cationic agents are surface-active and thus may act as dispersing agents. Two typical direct cotton dyes showed reduction in light fastness after treatment with cationic agents (Fig. 28 and 29), and the changes in their CFO curves and in the corresponding spectroscopic absorption curves (Fig. 30 and 31) are quite consistent with the reduction being due to dis-aggregation effects.

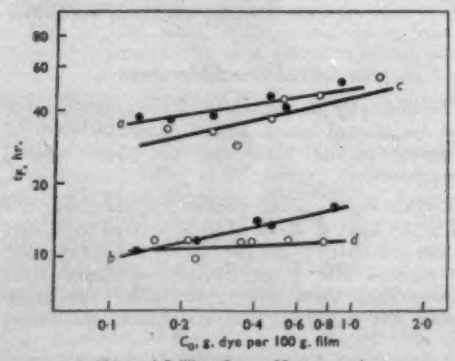


FIG. 29—CFO Curves for Direct Cotton Dyes on Cellophane Film

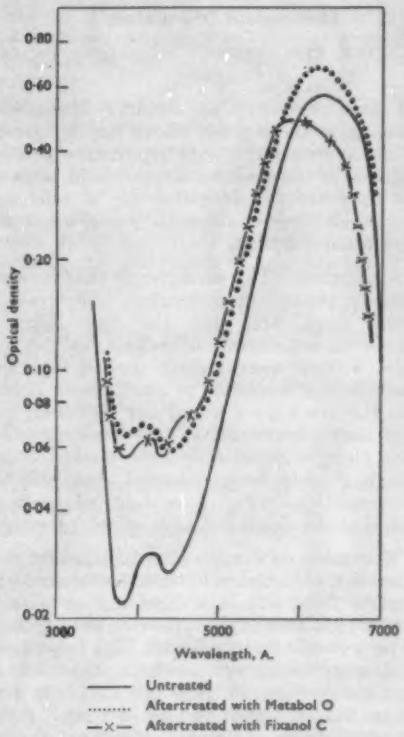


FIG. 30 — Light Absorption Curves for Chlorazol Sky Blue FF on Cellophane Film (Arbitrary dye concentrations)

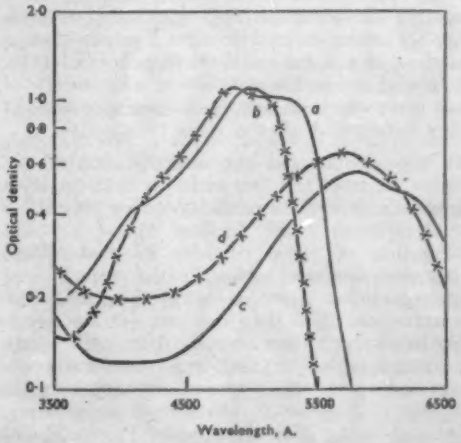


FIG. 31— Light Absorption Curves for Direct Cotton Dyes on Cellophane Film

Theoretical Discussion

ALTERNATIVE EXPLANATIONS OF SLOPES OF CFO CURVES

It is first necessary to consider alternative explanations to those given above for the nature of the CFO curves. The only alternative general explanations of the positive slopes would seem to be that they are due to either (*a*) a screening effect or (*b*) blockage of substrate pores, which are therefore discussed here.

(*a*) SCREENING—This would imply that the dyes are actually present in molecularly dispersed or monolayer form, and that the light fastness increases with concentration because, as the dye molecules become more tightly packed together, they become less accessible to irradiation. If this were so, the CFO curves would not be linear, but would be nearly horizontal at very low concentrations and then would steadily rise. Curves of this type have in fact been observed, but only in collodion solutions (Fig. 4 and 5), where it is probable that the dye is in a form of solid solution.

(*b*) BLOCKAGE OF PORES—The explanation here would be that, as the pores of the substrate become more nearly filled with dye, light and/or air and moisture are less able to reach the dye, even though it may be present as a monolayer. This hypothesis can be directly tested by considering the effect of changing the pore size of the substrate. If it were true, then the light fastness of a dye at a given concentration would obviously *fall* with increase in pore size of the substrate, whereas actually it increases (cf. Fig. 14, 15, 22–26).

It might still be true that, as suggested by Bean and Rowe[3], crystals are more resistant to light than are amorphous aggregates, which could account for the phenomena observed on soaping azoic dyes (Fig. 7 and 8) (an opposite effect— crystals being the more susceptible form— might explain the effect of soaping vat dyes (Fig. 10 and 11)); but this, of course, has no relation to the results of those tests which do not involve comparisions of fading before and after a given treatment.

It thus seems that the interpretation of the positive slope of the CFO curves is that the dyes are present as discrete particles (either crystalline or amorphous); and further that the size distribution of these particles is non-uniform in the sense described earlier, i.e. the proportion of larger particles increases with the total dye concentration. This does not rule out the possibility that some proportion of the dyes (particularly the water-soluble dyes) may be present as a monolayer; in fact, this must almost certainly be so in the case of the direct cotton dyes in cellulose, according to the work reported by Preston[23] and Morton[20]. This may account for the low slope of the CFO curves of some direct cotton dyes (e.g. Fig. 24). (Morton worked with ramie, which, being highly crystalline, may be less likely to encourage the development of aggregation than is the more amorphous regenerated cellulose used here.)

There is finally the argument that any aggregates present may arise from the effect of the heat generated during irradiation [cf. 6]. Heat would, however, tend directly to break up aggregates rather than form them. Even if heating due to irradiation indirectly assisted aggregates to form by driving off more adsorbed moisture from the substrate, some aggregates must have been present beforehand.

VALIDITY OF THE THEORETICAL MODEL

The validity of the theoretical model is demonstrated e.g. by the facts that all the CFO curves so far obtained have slopes between 0·01 and 0·80: i.e. they all lie between the theoretical limits of 0 and + 1·4; that dispersions of known uniform size distribution (System A) give CFO curves of (or nearly) zero slope; and that such curves make greater intercepts on the *y*-axis as the average particle size increases, whether the particle-size distribution is uniform or not (cf. Fig. 7 and 8).

THE RATE-AFFECTING FACTOR IN FADING

The most probable factor affecting the rate of fading is the surface area/weight ratio of the dye. The most likely alternative factor is the porosity of the substrate, which controls the rate of diffusion of the essential oxygen and water to the sites of photochemical reaction. The present work, however, seems to prove that porosity is not a direct controlling factor: if it were so, an increase in pore size would increase the rate of fading, whereas in fact it usually has the opposite effect.

SIGNIFICANCE OF THE SLOPES OF THE CFO CURVES

Many of the values for the slopes of the CFO curves lie between those for Systems B and C, implying that with increase in concentration the total dye surface increases more rapidly than does the photochemically active surface. Two reasons may be suggested for this— (*a*) the faces of the dye crystal are not all equally reactive, and (*b*) the crystals grow so that they block the pores of the substrate and only the dye surfaces at the "open" ends of the pores are in contact with air and water vapour.

Practical Considerations

The normally measured light fastness of a dye must be several times higher than the intrinsic resistance of its molecules to photochemical decomposition*.

In general, the more porous the substrate the higher the light fastness of dyes applied to it, and the less porous the lower the fastness. For example, tin-weighted silk gives dyeings of lower light fastness than does unweighted silk[14], probably because the weighting material blocks the pores.

* A factor even of four would represent only a small degree of aggregation. Consider e.g. an imaginary dye molecule forming a cube of linear dimensions 10 × 10 × 10 A.; if 64 such molecules coalesce to form a cube of dimensions 40 × 40 × 40 A., the weight increases by a factor of 4 compared with the surface area, so that the light fastness will increase by a similar factor. Even so, the linear dimensions of this particle are only about one-hundredth of those required for visibility in an optical microscope.

High light fastness at low depths of colour will be more readily obtained by the use of pigment dispersions than by dyeing processes. The reason is that dispersions should have the same particle-size distribution at all depths of dyeing (cf. System A, p. 219), and so the actual light fastness will not fall with reduction in concentration, but will remain constant and always above the intrinsic value for the individual dye molecule. Dyes applied from a solution (even when subsequently insolubilised, e.g. azoic or vat dyes) usually give CFO curves of positive slope; i.e. as their concentration is reduced the light fastness falls, and in the limit it must reach the intrinsic fastness of the individual dye molecule.

Drying conditions may influence the light fastness of dyed fabrics. (Data in Table VI of the previous paper[6] show variations attributable to this cause.)

Experimental

DYES

Most of the dyes used, and the samples of Fixanol C (ICI) and Metabol O (ICI), were of normal commercial quality. The samples of Chlorazol Sky Blue FF (*C.I.* 518), the *mero*cyanine dyes, and the azo-β-naphthol dye were pure laboratory preparations. The basic dye and its two lakes were supplied by James Anderson & Co. (Colours) Ltd. as, respectively, Victoria Blue BO 110%, a lake from an exact precipitation with tungstophosphoric acid, and a lake from a precipitation with a 25% excess of the complex acid.

FILMS AND FABRICS

Most of the regenerated cellulose films used were normal commercial Cellophane (thickness 0·001 in.). Those used for examining the effect of pore size were (*a*) dry "PT 600 cellulose film" and the equivalent 600 and 300 qualities in gel form. The dry film contained a water-soluble plasticiser, but this would be removed in the dyeing process. The aged gel films were stored wet in polythene bags in the dark at room temperature for about six months.

The viscose rayon materials used for the results in Fig. 20, 24, and 27 comprised three fabrics identical in structure (weave, plain; warp and weft, 1/18s (cotton count); set 60 × 54) but made from Fibro (Courtaulds) staple fibre of different physical characteristics, as follows—

(*a*) Standard Fibro (1·5 denier, $1\frac{7}{16}$ in. staple)

(*b*) Strong Fibro (1·25 denier, $1\frac{1}{4}$ in. staple), stated to be a Tenasco (Courtaulds) type of material, having a small crystallite size and thus a rather refined structure

(*c*) Low-swelling Fibro (1·5 denier, $1\frac{7}{16}$ in. staple), made from the same spinning as (*a*) but distinguished by having been recrystallised by a physical aftertreatment. It was stated that the X-ray diagram should be almost completely of cellulose IV type.

The imbibition values were — (*a*) 87%, (*b*) 86%, (*c*) 55%. These figures refer to moisture retention in a standardised test used by the manufacturers, and may be taken as a qualitative measure of the relative internal pore volume of the fibres.

The materials used for the results of Fig. 18, 19, 21–23, and 25 were taffeta fabrics made from continuous-filament yarns and all were identical in structure (weave, plain; warp and weft, 30 denier; set, 104 × 104; 0·8–0·9 oz./sq.yd.; ordinary viscose rayon and Tenasco, 12 filaments per yarn; Durafil (Courtaulds), 100 filaments per yarn). The respective imbibition values were 118, 94, and 69%.

All these fabrics were supplied commercially clean and scoured, and were used without further pretreatment.

The nylon film was clear transparent nylon sheet of 0·003 in. thickness.

The gelatin was of pure photographic quality, cast in films as described previously[6].

The collodion films used with the *mero*cyanine dyes were prepared by air-drying Necol (ICI) varnish, to which acetone solutions of the dye had previously been added.

The cast films were dried by placing the glass plates on a levelled metal sheet heated by four 130-w. carbon-filament lamps set at the corners of a 6-in. square with their filaments about 3 in. below the sheet. This arrangement was found to give rapid and even drying of the films.

The basic dye and its lakes were exposed in films of Lorilleux & Bolton's Middle Letterpress Litho Varnish, which is a heat-thickened linseed-oil varnish, of viscosity 70 poises at 25°C.

PREPARATION OF *MERO*CYANINE AND AZO DYE DISPERSIONS

The two *mero*cyanine dyes were—

2-(2-*n*-Hexadecyloxy-5-oxothiazolin-4-ylidene-ethylidene)-3-methylthiazolidine (I)

4-(3-Ethylbenzothiazolin-2-ylidene-prop-2′-ylidene)-2-ethylthiothiazol-5-one (II).

Dye I was treated as follows—

(*a*) Ground to a fine dispersion in water alone, then added in measured proportions to 6% aqueous gelatin solution, or 4·5% aqueous Cellofas A (ICI) (methyl ethyl cellulose) solution, with good agitation, before setting. These films had the coarsest particles.

(*b*) Added in measured proportions from a 0·03% solution in acetone to a 6% aqueous gelatin solution with good agitation, before setting. This gave slightly turbid films in which the particles were too small to be seen at 250× magnification.

(*c*) As (*b*), but in place of gelatin solution a mixture of 80 c.c. Necol (cellulose nitrate) varnish and 20 c.c. acetone was used. This gave completely transparent films in which no particles could be seen under the microscope.

Dye II was treated by methods (*a*) and (*b*), but additional series of films were prepared by incorporation of a separated precipitate (fine needles) made

by pouring an acetone solution into water and by separating particles of different sizes by elutriating the water-ground suspension. Unfortunately, only the finest of the dispersions showed any measurable fading over a long period (2–3 months) of continuous irradiation, and therefore no comparison could be made between the remainder.

The azo dye (*p*-anisidine→*β*-naphthol) was treated by methods (*b*) (in Cellofas A) and (*c*).

PREPARATION OF AZOIC-DYED CELLOPHANE FILMS

Sheets of Cellophane film (6 in. × 4 in.) were worked for periods varying from 1 to 15 min., in a flat dish at room temperature, in a solution containing, per litre, 2 g. of the Brenthol component and a total of 1 g. of sodium hydroxide, 2 g. of sodium chloride, and a little wetting agent (Calsolene Oil HS (ICI)), prepared in the normal manner. They were then removed, well rinsed, first in sodium chloride solution and then in water, and worked for 10 min. in a cold 0·05% solution of the diazotised base (neutralised with sodium acetate after diazotisation). One half of each piece was then boiled for 1 hr. in a solution containing 2·5 g. soap flakes and 2·5 g. sodium carbonate per litre. Both halves were finally well rinsed in water, oven-dried at 100°C., and mounted for irradiation.

PREPARATION OF DYED NYLON FILMS

Strips (4 in. × 3 in.) of nylon film were first scoured at 40°C. for 15 min. in a 0·5% solution of a non-ionic detergent (Lissapol N (ICI)), then rinsed, and dyed in a bath prepared as follows[21]— 0·1 g. of the vat dye powder, 14 c.c. of 20% aqueous sodium hydroxide solution, and 200 c.c. of water were heated to 80°C., then 2 g. of Formosul G (Br) and 0·2 c.c. of Dispersol VL (ICI) were added, and the temperature raised to 90°C. and kept thereat for 10 min. to ensure complete reduction. The nylon strips were dyed (lightly rolled, in test tubes) for varying periods of time, to obtain different depths of colour, then rinsed, air-oxidised, soaped at 60°C. for 15 min. in a soap solution (10 g./litre), and rinsed again. One half of each strip was then treated at 50°C. for 5 min. in 500 c.c. of a 1·5% solution of cinnamic acid in a 25% aqueous solution of acetone, next rinsed in hot water, soaped at 60°C. for 15 min. in a solution containing 10 g. soap and 0·5 g. sodium hydroxide per litre, and rinsed. Finally, all the strips were oven-dried at 100°C.

PREPARATION OF DIRECT- AND VAT-DYED CELLOPHANE FILM

The direct dyes were applied to Cellophane from hot water alone, the different depths of colour being obtained by dyeing rolled strips (10 in. × 1·5 in.) for different periods of time (1–15 min.) at 80–90°C. in 400 c.c. of dye solution (0·75 g./litre). The aftertreatments with Fixanol C and Metabol O were made by working the dyed films for 15 min. at 40°C. in a solution of 6 g. of reagent per 100 c.c., then well rinsing, and drying at 100°C. Metabol O was first recrystallised from water, with charcoal clarification.

The indigoid vat dyeings were prepared as follows— 20 c.c. of a 1% suspension of the dye powder was vatted at 60–70°C. for 10 min. after addition of 7 c.c. of 10% aqueous sodium hydroxide and 0·3 g. of sodium hydrosulphite. The vat was then diluted to 200 c.c. with warm water containing 1 c.c. of 10% sodium hydroxide and 0·1 g. of sodium hydrosulphite and maintained at 50–60°C. Strips of Cellophane (10 in. × 1·5 in.) were immersed for varying periods of time to give a range of depths, then rinsed, and air-oxidised. One half of each strip was then boiled for 15 min. in soap solution (1 g./litre), rinsed, and oven-dried.

All these films were clipped to a rectangular spacer of glass rod during dyeing to ensure levelness.

DYEING OF VISCOSE RAYON FABRICS*

The viscose rayon fabrics were dyed in 1·5-g. portions for 45 min. at 85–90°C. in 40-volume baths containing 0·25–6·0% dye (referred to weight of fabric), 20% Glauber's salt, and a few drops of wetting agent (Dispersol VL), then well rinsed, and oven-dried. The amount of dye on each of the samples of the first series was measured by absorptiometric determination of the liquors after dyeing, and on those of the second series by dissolution of the fibre in aqueous sulphuric acid.

PREPARATION OF DYED CELLULOSE ACETATE FILM

Disperse dyes were applied by the normal process to clear 0·003 in. unplasticised cellulose diacetate film.

PREPARATION OF DYED ANODISED ALUMINIUM

Pieces (6 in. × 4 in.) of aluminium foil (0·002 in. thick, 99·99% purity) were anodised for 1 hr. in either (*a*) 20% (wt./vol.) sulphuric acid at 25°C. and 12 v. or (*b*) 3% aqueous chromic acid at 40°C. and 45 v., the electrolyte being continuously agitated. The foil was then well rinsed, dried at 130–140°C. for 1 hr., and stored in a desiccator until ready for use. The actual weight of film present was determined by weighing the anodised material before and after dissolving the film in chromic acid–phosphoric acid solution. Discs 1 in. in diameter, to fit the spectrophotometer reflectance attachment, were cut from the finished sheets, and dyed in 10-c.c. portions of dye solutions of concentration 0·0125–0·10 g./litre for 10 min. on a boiling water bath. They were then rinsed, dried, and clamped between glass for irradiation, the back being shielded from the light. When measurements were complete on each series on one side of the discs, they were reversed and a second set of fading-rate curves determined for the other side. The two sets of readings are plotted individually in Fig. 26. Owing to the difficulty of anodising the two sides uniformly, the depth of dyeing was not the same on the two sides. For this reason also, it is not practicable to correlate the data for the total amount of dye adsorbed by each disc (calculated from absorptiometric measurements of the exhaust liquors) with the individual reflectance measurements for the two sides. Consequently the ordinates of Fig. 26 refer to a 10% increase of reflectance values, and the abscissae to the initial reflectance readings for each individual disc side. The actual

* Microscopic examination of fibre cross-sections showed dye penetration to be complete.

initial percentages of (commercial) dye present (referred to the total weight of film on both sides of each disc) are given in Table III*.

TABLE III

Initial Depths of Dyeing on Anodised Aluminium Discs

(% commercial dye on total wt. of film on both sides of disc)

Coomassie Brilliant Blue G 250		Coomassie Violet 2 RS	
H_2SO_4 Film	H_2CrO_4 Film	H_2SO_4 Film	H_2CrO_4 Film
0·44	0·23	0·18	0·025
0·58	0·6	0·42	0·13
0·98	0·85	0·66	0·18
1·8	1·5	—	0·21

CREASE-RESIST TREATMENT cf.[15]

Two solutions were first prepared—

(*a*) 200 c.c. of formaldehyde solution (40%) was neutralised to pH 7 with 1% sodium hydroxide, using B.D.H. Universal Indicator externally; 100 g. urea was then added, and when it had dissolved, 9 c.c. ammonia solution (sp.gr. 0·880)

(*b*) Ammonium dihydrogen phosphate (9 g.) was dissolved in 50 c.c. water and neutralised to pH 7 with 10% ammonia solution

Solution (*a*) was boiled under reflux for 3 min., and cooled rapidly in running water to below 20°C. Solution (*b*) was then added, and the mixture diluted with cold water to 20° Tw. strength. The dyed fabric (standard Fibro) was padded in the cold mixed solution, blotted between filter paper, baked at 130°C. for 3 min., soaped in a solution containing 2·5 g. soap flakes and 2·5 g. sodium carbonate per litre for 5 min. at 50°C., then rinsed, and oven-dried.

VAT DYE IN GELATIN

Samples of Caledon Brilliant Orange 6R pastes of different specified average particle sizes (obtained by courtesy of Imperial Chemical Industries Ltd., Dyestuffs Division) were dispersed in gelatin and irradiated in the hope that some exact relation could be obtained between fading rate and particle size, but the experiment had to be abandoned on account of the very low rate of fading even of the finest particles.

DETERMINATION OF CONCENTRATION IN SUBSTRATES

The amount of colouring matter in the films used in the experiments with azo and *mero*cyanine dyes was calculated directly from the volume of the portions of dispersion added to the substrate, and was therefore an absolute value in each case. In other systems, the relative concentrations of dye in the films of each series were determined by optical density measurement at the wavelength of maximum absorption. (The absolute concentration values are not required.) Examination of a series of *mero*cyanine dye dispersions showed that Beer's law is obeyed for all concentrations of a dispersion of the same average particle size, but the aggregated systems formed on drying uniformly dyed films do not, as already discussed, have the same average particle size at all concentrations. Hence it was necessary to establish that the dye concentration values obtained for untreated and treated films in any given series are strictly comparable between the series. It was considered possible that aggregation might alter the relation between concentration and optical density sufficiently to affect the relative position of the curves for treated and untreated samples.

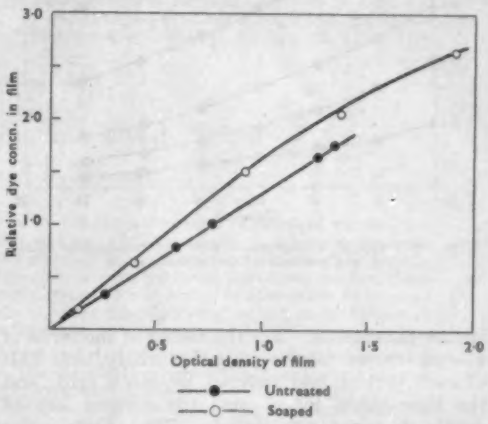


FIG. 32 — Relation between Dye Content and Optical Density of Azoic-dyed Cellophane Films (*m*-Chloroaniline→β-naphthylamide of 2:3-hydroxynaphthoic acid)

Accordingly, a test was made with samples of azoic-dyed films, these being chosen because they represent the most extreme case of visible change in aggregation between treated and untreated materials. Samples of film of the same dimensions from the soaped and unsoaped series were taken, and their respective optical densities recorded in film form and again in solution form after each film had been dissolved by leaving to stand for a few hours in the refrigerator in a given volume of sulphuric acid (80 c.c. H_2SO_4 (sp.gr. 1·84) + 20 c.c. water). The results (Fig. 32) show that there is a slight difference between the curves for the two series, but the error introduced by this cause is very much less than the effects of the aftertreatments on the light fastness itself. It is therefore assumed that no significant difference due to this cause need be feared in any of the other series.

IRRADIATION PROCEDURE AND MEASUREMENT OF FADING RATES

Fuller details of these are given in the previous paper[6]. The exposures were made to the direct light of a 400-w. Osira (General Electric Co. Ltd.) mercury-vapour lamp. The fading rates were measured by determining the reduction in optical density of the films at regular intervals during irradiation, at the wavelength of maximum absorption, on a Unicam SP 500 photoelectric

* It was found that dyeing for much longer periods had no significant effect on fastness.

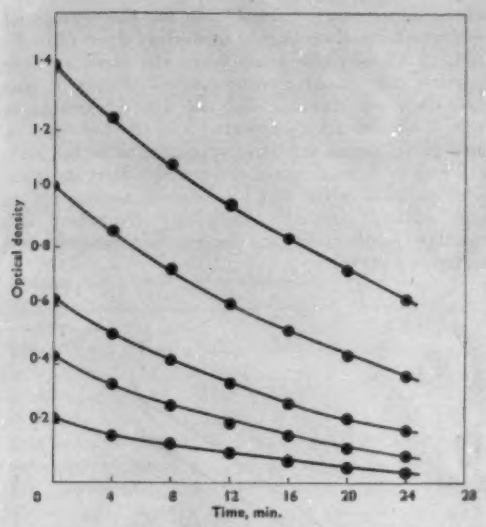


FIG. 33 — Typical Fading-rate Curves for Transparent Films (*mero*Cyanine Dye I in Collodion Film)

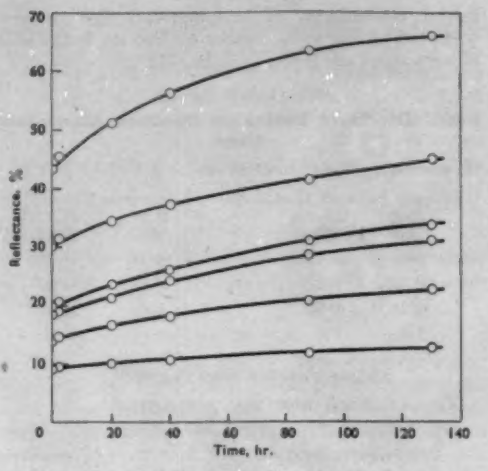


FIG. 34 — Typical Fading-rate Curves for Fabrics (Chloranol Sky Blue FF on Normal Viscose Rayon Fabric)

spectrophotometer. For the fading of the series of viscose rayon fabrics judged visually, one half of each portion was covered by black card, and the time taken for a small but distinct loss of depth to occur was noted. The other series of viscose rayon fabrics (Fig. 18–25) and the pieces of anodised aluminium were cut in 1-in. circles suitable for insertion in the spectrophotometer reflectance attachment.

The fading-rate curve for each sample was plotted usually until about 25% of the original dye had faded, and t_F was measured from this curve, being the time required for a loss of 10% of the *initial* amount of dye present, measured either from the start of irradiation or, in those cases where the fading rate shows initial irregularities cf. [6], from the earliest point at which it is regular in all the curves of the series. Fig. 33 and 34 show two typical groups of fading-rate curves, for sets of transparent films and viscose rayon fabrics respectively.

The computation of the CFO curves shown in the present paper has necessitated the determination of about 400 fading-rate curves.

All optical density and reflectance measurements were made on a Unicam SP 500 photoelectric spectrophotometer.

Appendix

CALCULATION OF THEORETICAL SLOPES OF CFO CURVES FOR THE MODEL SYSTEMS B AND C

System B

It is assumed that the total loss of dye in a given period is proportional to the total surface area of the cube and that the photochemical reaction at the surface is of the first order. The amounts lost from each of a series of cubes, e.g. of initial weights 2, 4, 5, and 8 units, when 10% has been lost by a cube of unit weight, are thus respectively 7·95, 6·3, 5·85, and 5·0% of the total, and the corresponding times for 10% loss (t_F) can then be determined from the first-order relation. This enables the CFO curve to be plotted.

System C

A graphical method is used to determine t_F for a 10% fade; assuming, say, 0·1 unit loss in 1 hr. from unit weight, the first-order relation is used to determine the times required for the loss of 0·2, 0·3, etc. unit, these then being the corresponding t_F values for initial weights of 2, 3, etc. units.

* * *

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The Effect of Chlorine on Dyed Nylon

A. N. DAVIDSON and R. PRESTON

After dry chlorination of wool, the chlorine retained by the fibres is present as hydrochloric acid (i.e. is extractable by alkaline solutions); whilst with nylon it is shown that the chlorine is more strongly combined, so that it is not completely extracted by sodium carbonate solution but can be removed by sodium bisulphite solution. Generally, absorption of chlorine by nylon is attended by an increase in fluidity of the nylon in *m*-cresol and a corresponding decrease in tensile strength; the degree of absorption of chlorine appears to vary with the type of dye used. The presence of wool has a "buffering" action on the uptake of chlorine by nylon and hence tends to protect the nylon from excessive damage.

Of the groups of dyes examined (chrome, level-dyeing and neutral-dyeing acid dyes, together with a few soluble acetate-rayon dyes and direct cotton dyes), the chrome dyes were satisfactory in their fastness to chlorination. The lighter shades of neutral-dyeing acid dyes were useless and the other dyeings occupied intermediate positions. All these dyes, with a few exceptions, have a slightly higher fastness to wet chlorination.

The above observations are illustrated in detail by tabulated and graphed data, together with colorimetric data for a representative selection of the dyes before and after dry chlorination.

Introduction

The admixture of nylon with wool has raised many problems for the dyer and finisher. One specific problem, described by a finisher who uses the dry (gaseous) chlorination process [1], concerned the effect of chlorine on the colour of dyed nylon used as a splicing thread in ingrain half-hose. During processing it was noticed that, in parts of the charge, the colour of the nylon was changed to varying degrees and in an apparently random fashion. Further, the actual dyes used were scheduled as fast to chlorine when dyed on wool. During discussions, it was learnt that the problem arises not only with the dry chlorination process, but also with goods treated by wet chlorination, i.e. by chlorine or hypochlorite ions in solution. It seemed, therefore, that a useful purpose would be served by investigating the problem, and it was hoped that in addition information would be obtained regarding the general reaction between chlorine and nylon.

It was decided at the beginning to use the dry chlorination process for the bulk of the work, since this technique offers the greatest degree of control and, further, the products of the reaction remain on the fibre after chlorination, so that more analytical data may be obtained than by any other chlorination method. To complete the work, typical samples of the dyed nylon were treated by a wet chlorination technique and also with alkaline hydrogen peroxide as another oxidising agent.

Materials

(i) NYLON— This was obtained in the form of staple-fibre yarns— (*a*) undyed, 1/50s worsted counts; and (*b*) dyed, 2/24s worsted counts.

(ii) WOOL— Undyed, hand-knitting yarn (3/18s worsted counts from 64s wool), soap-scoured, was used.

The dyed nylon was machine-knitted into a plain stockinette fabric with the wool, 1 end of nylon and 1 end of wool being fed simultaneously to the machine. The nylon in the fabric was approx. 33⅓% by weight, i.e. a similar proportion to that present in the heels and toes of the spliced half-hose.

Treatments

(*a*) DRY CHLORINATION

These experiments were carried out in a 1000-ml. round-bottom flask, fitted with an inlet and an outlet tube incorporated in a ground glass joint. Approx. 60 g. of wool–nylon fabric or 30 g. of nylon yarn (dyed or undyed), previously dried to a regain of 5% and 2% respectively, was placed in the flask, which was sealed and almost completely immersed in a water bath maintained at the desired temperature. After 1 hr. the flask was evacuated to a vacuum of 27 in. of mercury, and then dry chlorine gas admitted until the vacuum fell to 15 in., corresponding to approx. 1·5 g. of chlorine. After 1 hr. the flask was opened and the fabric or yarn removed. Since it was found

that, unlike wool, nylon after dry chlorination retains "active" chlorine, i.e. it will liberate iodine from acidified potassium iodide solution, the chlorinated samples were treated immediately on removal from the flask by soaking in a solution containing either 2% of sodium sulphite or 2% of sodium bisulphite (both calculated as the anhydrous salts on the weight of the material) at 20°C. for 20 min., then rinsing in water, and drying. The dried fabric was dissected into wool and nylon yarns by a continuous mechanical method, and the nylon wound on to a cone for testing.

(*b*) WET CHLORINATION

Approx. 10 g. of wool–nylon fabric was wetted out in 500 ml. of water, to which a small amount of Alcopol O* was added to assist penetration, and treated with 3% of chlorine (on the weight of the fabric) by running in simultaneously sodium hypochlorite and hydrochloric acid from two burettes. The rates of flow of the liquids were adjusted so that both solutions entered completely in exactly 20 min., whilst the amount of acid was chosen to maintain the pH at 2. The chlorinated fabric was cleared and finished as described in (*a*), using 2% sodium bisulphite calculated on the weight of the fabric.

(*c*) HYDROGEN PEROXIDE TREATMENT

Samples of dyed nylon yarn were treated with hydrogen peroxide of 10 volumes strength, containing 0·5% of sodium pyrophosphate. The liquor : yarn ratio was 20 : 1. The temperature was maintained at 45° ± 1°C. for 60 min., the samples then being rinsed and dried.

Testing Methods

1. ESTIMATION OF CHLORINE AND CHLORIDE

(*a*) *Total Chlorine*

A modified open-Carius[2] method was employed. One gram of material was thoroughly wetted out with a known quantity of 0·1 N. silver nitrate in a Kjeldahl flask and boiled with 10 ml. of fuming nitric acid until the solution became pale brown, when 40 ml. of a saturated solution of potassium permanganate was added. The boiling was continued until the solution was colourless. The precipitated silver chloride was filtered off on a sintered glass crucible (grade 4), and the residual silver nitrate estimated by titration with standard potassium thiocyanate, using ferric ammonium sulphate in dilute nitric acid as indicator.

(*b*) *"Free" Chlorine*

The material (1·5 g.) was shaken with 75 ml. of a solution containing 3·5 g. of sodium carbonate (anhydrous) per litre for 60 min., and the chloride estimated in a 50-ml. aliquot, acidified with nitric acid, by the Volhard method, viz. addition of excess of standard silver nitrate, filtration, and back titration with standard potassium thiocyanate.

(*c*) *"Combined" Chlorine*

After extraction of the "free" chlorine, the sample was washed in distilled water, and the total remaining chloride estimated by the open-Carius method, (*a*) above.

* Allied Colloids (Manufacturing) Co. Ltd.

2. BREAKING LOAD AND EXTENSION AT BREAK

The nylon yarn was conditioned for 24 hr. at 65% R.H. and 21°C., and successive 10-in. lengths were tested on the Baer single-thread machine, to which was fitted an electromagnetic stop.

3. FLUIDITY

The method used was essentially that described by Boulton and Jackson[3]. Nylon (0·4 g. dry weight) was dissolved in 10 ml. of *m*-cresol by shaking overnight, and the solution transferred to the viscometer jacket, which stood in a thermostat maintained at 20° ± 0·2°C. (the temperature was changed to 25° ± 0·2°C. for the testing of the dyed nylon samples on account of the difficulty of maintaining the lower value during the summer months). The time of flow of the solution was measured to within 0·5%, and the instrument constant K determined with a standard solution of glycerol. Since it was found that the nylon solutions became degraded on standing, whether in direct or in diffused daylight, all determinations were made on freshly prepared solutions. The results were expressed as fluidity units, i.e. reciprocal poises × 100.

4. ESTIMATION OF COLOUR CHANGE

Two methods were used to estimate the colour change which took place during the treatments, viz. (*a*) depending on the visual assessment, and (*b*) a quantitative method involving measurement of the percentage of light reflected from the samples at certain fixed wavelengths.

(*a*) *Visual Assessment*

Samples of the yarns were carefully wound on to white cards, and the degree of alteration of colour was estimated by the method described by Messrs. Imperial Chemical Industries Ltd.[4]. The majority of samples lost depth of colour, but when a change of hue occurred simultaneously an additional penalty, which did not exceed two units, was imposed.

(*b*) *Quantitative Method*

The principles underlying the measurement of colour by the C.I.E. primaries X, Y, Z are well known, and need not be given here[5, 6].

In the present experiments, the instrument used was the Cambridge Spectrophotometer SP 500, fitted with the Diffuse Reflectance Unit. The samples used in § 4(*a*) could not be used, as the holder for the sample on the reflectance unit required a circular pattern. Small patterns were therefore made in a plain weave from a representative group of the original yarns and the corresponding dry-chlorinated samples, on a Speed Weve darning machine[7] having 22 ends and picks per inch, and samples approx. 1 inch in diameter were cut from them. The determination of the factors X, Y, Z ("tristimulus values") when the sample is illuminated by C.I.E. Standard Illuminant C was made by using the 10 selected ordinate method[8].

The chromaticity and the overall reflectance values calculated from the experimental data are given in Table II.

Results

1. DRY CHLORINATION OF UNDYED NYLON ALONE

Extraction of chlorinated wool with sodium carbonate solution removes all the chloride present, and the figures obtained for chloride absorption by the open-Carius and the sodium carbonate extraction methods are equal. It is not so with nylon, for chlorine may be detected in chlorinated nylon that has been extracted with sodium carbonate, but extraction with sodium bisulphite removes all the chlorine.

The use of other alkaline solutions (0·4% caustic soda or 2% sodium sulphite at pH 8) gave analytical figures similar to those afforded by extraction with sodium carbonate. These results suggest that chlorine is chemically bound to, or strongly absorbed by, the nylon, the resulting attachment being sufficiently strong to withstand alkaline solutions. The chlorine absorbed by the nylon may therefore be subdivided into *free*, i.e. that which is extractable by aqueous alkaline solutions, and *combined*, that which remains on the nylon after alkaline extraction.

A further difference between nylon and wool is that nylon smells strongly of chlorine after removal from the chlorination chamber, even after it has been allowed to stand for a period in the air. This indicates that gaseous chlorine is adhering to the surface of the fibres, and thus the free chlorine consists of elemental chlorine together with hydrochloric acid formed by the reaction of the chlorine with the nylon (when part of the chlorine is apparently combined with the nylon).

Examination of the nylon after extraction with sodium bisulphite failed to show the presence of sulphur, indicating that the extraction of combined chlorine does not take place by the replacement of chlorine by a sulphur-containing group.

A series of chlorinations was made using a constant amount of chlorine gas at a fixed temperature and varying the time of treatment from 20 min. to 60 min. Under these conditions, it was not found possible to obtain a total chlorine absorption higher than 0·77%. To obtain higher chlorine absorptions, a further series was treated in which the amount of chlorine admitted and the time of treatment were kept constant and the temperature varied from 20° to 65°C. The chlorine absorptions (total, free, and combined) and the fluidities of the chlorinated yarns were determined, and the yarns tested for breaking load and extension at break. The relations between rise in fluidity, chlorine absorbed, and percentage loss in strength at break are shown in Fig. 1 and 2.

2. DRY CHLORINATION OF DYED NYLON ALONE AND IN ADMIXTURE WITH WOOL

Since the main object of these experiments was to determine the effect of chlorine on the dye, it was decided that only a random sample would be used for the investigation of the effect of chlorine on the strength at break, and fluidity measurements would be made on all the samples chlorinated at the lower temperature.

The chlorine absorptions and fluidity results are given in Table I, and the data from the random samples are represented in Fig. 3 and 4. In Fig. 3 the points marked are the mean combined chlorine contents and mean fluidity rises for the groups of dyes, the solid line giving the best representation for all the figures obtained. Over 90% of all the results fall within the broken lines. In Table I the dyes are grouped according to the type of dye, and the order in each group follows the classification obtained for the colour change, these assessments and the colorimetric data being given in Table II. The usual notation for the assessment of colour change is employed, viz. "5" represents no change and "1" the greatest change. Where an alteration in hue occurs, as distinct from loss of depth of colour, this is indicated, e.g. "towards red". In the colorimetric data, the x, y, and Y values have been calculated from the reflectance data by the method given in the *Handbook of Colorimetry*[8].

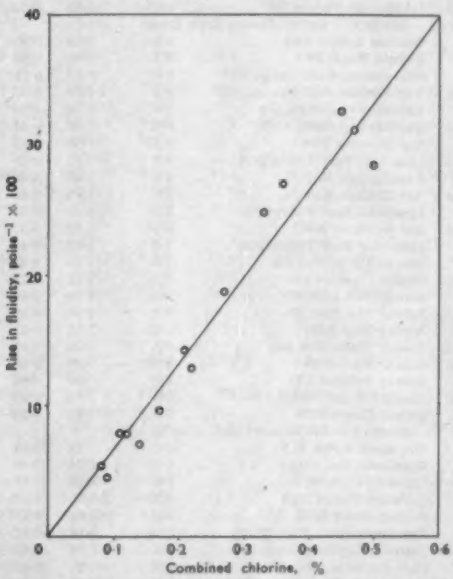

FIG. 1 — Relationship between Combined Chlorine and Fluidity Rise (Undyed Nylon)

The mean chlorine absorptions for the samples chlorinated at 50°C. are given in Table III.

3. EFFECT OF WET CHLORINATION ON THE COLOUR OF DYED NYLON

A random selection of the yarns were wet-chlorinated both in the presence of wool and alone. The results for the assessment of colour change are given in Table IV, figures for the fastness to wet chlorination on wool being included for comparison.

4. EFFECT OF HYDROGEN PEROXIDE ON THE COLOUR OF DYED NYLON

A random selection of the yarns were treated with hydrogen peroxide, the results being given in Table V.

TABLE I

Chlorine Absorption and Fluidity of Dyed Nylon

No.	Dye (ICI)	Depth (%)	Chlorine Absorption (% by wt.) at 27°c. Chlorinated with Wool			Chlorinated Alone		Fluidity (reciprocal poises × 100) Unchlorinated	Chlorinated with Wool		Chlorinated Alone	
			Free	Combined	Wool	Free	Combined		Fluidity	Fluidity Rise	Fluidity	Fluidity Rise
	Undyed	—	0·71	0·12	0·92	1·20	0·49	60·20	77·00	16·80	84·70	24·50
	GROUP A— CHROME DYES											
1	Solochrome Black WDFA	10·0	0·92	0·27	1·06	1·00	0·47	45·62	50·41	4·79	53·69	8·07
2	Solochrome Black 6BNS	10·0	0·72	0·28	0·91	1·24	0·59	54·63	62·76	8·13	78·03	23·40
3	Solochrome Maroon WN	8·0	1·05	0·33	1·05	1·22	0·59	58·89	69·88	10·99	77·23	18·34
4	Solochrome Brown RHS	1·0	1·07	0·28	1·12	1·28	0·50	54·33	63·97	9·64	70·73	16·40
4(a)	Solochrome Brown RHS	0·2	0·96	0·28	1·00	1·23	0·51	57·32	68·37	11·05	79·02	21·70
5	Solochrome Flavine GS	0·75	1·16	0·27	0·98	1·50	0·61	53·01	66·10	13·09	73·19	20·18
6	Solochromate Fast Orange 2RS	1·0	1·23	0·16	1·10	1·09	0·47	60·50	79·11	18·61	81·96	21·46
7	Solochromate Fast Grey BS	1·0	1·42	0·27	0·98	1·82	0·70	59·88	69·74	9·86	83·77	23·89
8	Solochrome Yellow 2GNS	0·75	1·04	0·15	0·81	1·82	0·32	54·44	71·47	17·03	69·15	14·71
9	Solochrome Dark Blue BS	2·0	1·04	0·35	1·03	1·36	0·60	61·66	65·43	3·77	79·71	18·05
9(a)	Solochrome Dark Blue BS	0·4	1·14	0·35	1·11	1·52	0·63	57·40	70·81	13·41	83·43	26·03
10	Solochrome Navy Blue WN	4·5	0·89	0·27	0·84	1·26	0·52	45·72	53·01	7·29	58·53	12·81
11	Solochrome Brown WN	3·0	0·94	0·33	0·99	1·22	0·62	58·46	71·74	13·28	80·44	21·98
12	Solochrome Red ERS	1·0	1·22	0·38	1·01	1·53	0·71	58·72	73·26	14·54	81·14	22·42
12(a)	Solochrome Red ERS	0·2	1·33	0·28	1·07	1·34	0·55	56·20	69·00	12·80	73·35	17·15
7(a)	Solochromate Fast Grey BS	0·2	1·11	0·32	1·00	1·73	0·64	63·27	71·17	7·90	83·58	20·31
6(a)	Solochromate Fast Orange 2RS	0·2	1·30	0·27	0·92	1·18	0·57	56·08	66·68	10·60	79·05	22·97
5(a)	Solochrome Flavine GS	0·15	1·35	0·24	1·02	1·29	0·36	53·91	68·85	14·94	82·68	28·77
	GROUP B— LEVEL-DYEING ACID DYES											
13	Croceine Scarlet 3BS	1·5	1·09	0·40	0·86	1·25	0·64	66·74	88·24	21·50	91·37	24·63
14	Rufasol Black BS	3·5	0·90	0·33	0·99	1·19	0·56	70·81	81·28	10·47	89·06	18·25
15	Naphthalene Fast Orange 2GS	2·0	0·87	0·31	0·82	1·52	0·65	63·08	71·68	8·60	87·40	14·32
15(a)	Naphthalene Fast Orange 2GS	0·4	0·99	0·36	0·83	1·51	0·64	63·04	85·11	22·07	97·51	24·47
16	Naphthalene Orange GS	1·5	0·68	0·24	0·61	1·65	0·58	63·48	77·34	13·86	101·5	38·02
16(a)	Naphthalene Orange GS	0·3	1·33	0·41	0·97	1·54	0·74	64·21	87·18	22·97	103·3	39·09
19	Azo Geranine 2GS	1·25	1·00	0·33	0·80	1·24	0·48	66·92	80·95	14·03	93·33	26·41
20(a)	Lissamine Fast Yellow 2GS	0·2	0·95	0·38	0·92	1·38	0·65	64·80	84·70	19·90	101·0	36·20
17	Naphthalene Red JS	1·0	1·33	0·43	0·95	1·51	0·60	51·34	82·10	30·76	99·97	42·63
17(a)	Naphthalene Red JS	0·2	1·04	0·42	0·94	1·60	0·64	62·47	78·21	15·74	95·52	33·05
18	Lissamine Fast Violet 2BS	1·5	1·07	0·35	0·97	1·65	0·74	66·34	82·28	15·94	88·92	22·58
19(a)	Azo Geranine 2GS	0·25	1·02	0·38	1·00	1·40	0·58	66·99	79·47	12·48	87·68	20·69
20	Lissamine Fast Yellow 2GS	1·0	1·06	0·49	1·01	1·51	0·74	62·50	79·95	17·45	85·51	23·01
21	Solway Ultra Blue BS	1·5	1·70	0·34	1·08	1·68	0·81	64·15	83·68	19·53	91·24	27·09
13(a)	Croceine Scarlet 3BS	0·3	1·12	0·37	0·98	1·49	0·62	51·52	72·28	20·76	88·24	36·72
22	Solway Sky Blue BS	1·5	0·98	0·40	1·04	1·39	0·63	66·61	79·66	13·05	94·73	28·12
22(a)	Solway Sky Blue BS	0·3	1·24	0·45	1·05	1·43	0·63	65·36	85·53	20·17	90·24	24·88
23	Solway Blue BNS	1·25	1·12	0·41	1·00	1·85	0·76	66·99	80·02	13·03	93·07	26·08
21(a)	Solway Ultra Blue BS	0·5	1·06	0·47	1·10	1·50	0·90	64·45	83·08	18·63	91·37	26·92
23(a)	Solway Blue BNS	0·25	0·90	0·35	0·85	1·48	0·70	65·42	83·99	18·57	97·52	32·10
24	Solway Celestol BS	2·0	0·99	0·32	0·86	1·49	0·66	68·17	79·02	10·85	88·57	20·40
18(a)	Lissamine Fast Violet 2BS	0·3	1·16	0·45	1·01	1·72	0·79	65·48	83·68	18·20	92·66	27·18
24(a)	Solway Celestol BS	0·4	1·72	0·50	1·20	1·45	0·56	63·68	87·58	23·90	96·45	32·77
	GROUP C— NEUTRAL-DYEING ACID DYES											
25	Coomassie Green WN	10·0	1·14	0·39	1·16	1·15	0·56	63·63	72·80	9·17	77·57	12·94
26	Coomassie Red PGS	1·75	1·25	0·39	0·94	1·50	0·65	60·35	91·75	31·40	101·6	41·25
27	Carbolan Crimson BS	1·0	1·16	0·37	0·98	1·73	0·81	62·76	82·85	20·09	90·18	27·32
28	Carbolan Yellow 3GS	0·75	0·90	0·37	0·84	1·40	0·71	62·15	81·91	19·76	98·49	36·34
29	Solway Green GNS	1·0	1·05	0·42	1·00	1·57	0·80	61·89	84·12	22·23	109·1	47·21
30	Coomassie Navy Blue 2RNS	2·25	1·16	0·42	1·17	1·38	0·68	61·09	80·58	19·49	88·35	27·26
31	Carbolan Brilliant Green 5GS	1·5	1·13	0·48	0·89	1·44	0·87	60·24	79·82	19·58	82·79	22·55
32	Carbolan Blue BS	2·0	1·21	0·42	0·98	1·48	0·69	61·15	84·89	22·74	91·01	29·86
33	Carbolan Brilliant Blue 2RS	2·0	1·05	0·52	0·96	1·42	0·81	66·86	88·51	21·65	99·77	32·91
34	Carbolan Violet 2BS	2·0	1·39	0·50	1·30	1·72	0·89	66·92	89·37	22·45	94·48	27·46
34(a)	Carbolan Violet 2BS	0·4	1·31	0·21	0·82	1·56	0·50	60·13	77·82	17·69	81·14	21·01
35	Carbolan Green GS	1·5	1·20	0·47	1·10	1·55	0·77	61·94	88·28	26·34	106·3	44·36
30(a)	Coomassie Navy Blue 2RNS	0·45	1·09	0·50	1·06	1·43	0·78	60·88	83·58	22·70	97·52	36·64
32(a)	Carbolan Blue BS	0·4	1·06	0·39	0·90	1·62	0·73	55·11	78·83	23·72	100·3	45·19
29(a)	Solway Green GNS	0·2	1·30	0·50	1·11	1·73	0·87	61·01	82·52	21·51	90·51	29·50
33(a)	Carbolan Brilliant Blue 2RS	0·4	1·23	0·59	1·16	1·67	0·85	62·22	80·91	18·69	98·88	36·66
35(a)	Carbolan Green GS	0·3	1·15	0·53	0·95	1·60	0·76	61·55	82·69	21·14	91·77	30·22
36	Coomassie Brilliant Blue RS	2·0	1·10	0·39	0·97	1·55	0·76	65·40	84·70	19·30	88·02	22·62
36(a)	Coomassie Brilliant Blue RS	0·4	1·31	0·34	0·79	1·45	0·54	63·39	77·52	14·13	88·94	25·55
26(a)	Coomassie Red PGS	0·35	1·75	0·41	1·14	1·91	0·78	58·19	84·96	26·77	105 5	47·31
31(a)	Carbolan Brilliant Green 5GS	0·3	1·26	0·33	1·01	1·62	0·62	60·61	81·91	21·30	87·68	27·07
27(a)	Carbolan Crimson BS	0·2	1·23	0·46	0·96	1·52	0·76	56·66	83·33	26·67	101·5	44·84
28(a)	Carbolan Yellow 3GS	0·15	1·22	0·33	0·96	1·64	0·61	60·28	83·24	22·96	96·05	35·77
	GROUP D—SOLUBLE ACETATE-RAYON DYES											
37	Solacet Fast Green 2GS	4·0	0·90	0·32	0·94	1·13	0·78	63·46	82·73	19·27	93·11	29·65
38	Solacet Fast Orange 2GKS	1·5	1·17	0·36	1·02	1·51	0·59	64·45	80·67	16·22	86·38	21·93
38(a)	Solacet Fast Orange 2GKS	0·3	1·39	0·39	1·08	1·73	0·74	62·82	81·96	19·14	108·6	45·78
39	Solacet Fast Red 5BGS	1·25	1·16	0·44	1·00	1·49	0·73	61·55	87·72	26·17	96·50	34·95
39(a)	Solacet Fast Red 5BGS	0·25	1·25	0·34	0·93	1·61	0·62	63·06	80·60	17·52	89·00	25·92
37(a)	Solacet Fast Green 2GS	0·8	1·02	0·35	0·98	1·51	0·62	63·81	86·10	22·29	101·1	37·29
	GROUP E— DIRECT DYES											
40	Chlorazol Fast Red FS	1·5	1·04	0·43	0·99	1·41	0·83	66·92	81·72	14·80	95·92	29·00
40(a)	Chlorazol Fast Red FS	0·3	0·89	0·38	0·86	1·58	0·70	64·37	91·37	27·00	105·8	41·43

TABLE II

Fastness Assessment and Colorimetric Data

No.	Fastness Assessment after Chlorination		Change in Hue towards	Colorimetric Data								
				Unchlorinated			Chlorinated with Wool at 27°C.			Chlorinated Alone at 27°C.		
	With Wool	Alone		x	y	Y	x	y	Y	x	y	Y
Undyed	—	—	—	0·314	0·321	63·7	0·318	0·327	62·6	0·318	0·327	62·8
	GROUP A— CHROME DYES											
1	5	5	—	—	—	—	—	—	—	—	—	—
2	4–5	4–5	—	—	—	—	—	—	—	—	—	—
3	4	4	—	—	—	—	—	—	—	—	—	—
4	4	4	Red	—	—	—	—	—	—	—	—	—
4(a)	4	4	Red	—	—	—	—	—	—	—	—	—
5	4	4	—	0·425	0·445	33·7	0·410	0·430	34·2	0·404	0·421	38·2
6	4	4	—	0·492	0·383	25·8	0·486	0·383	25·4	0·489	0·383	25·6
7	4	4	Red	0·278	0·274	13·8	0·287	0·296	16·4	0·289	0·296	16·1
8	3–4	3–4	—	0·416	0·396	20·9	0·394	0·382	26·0	0·391	0·385	26·2
9	3–4	3–4	Green	—	—	—	—	—	—	—	—	—
9(a)	3	3	Green	—	—	—	—	—	—	—	—	—
10	3	3	Red	—	—	—	—	—	—	—	—	—
11	3	3	Red	—	—	—	—	—	—	—	—	—
12	3	3	—	0·438	0·266	11·6	0·421	0·275	12·3	0·415	0·267	13·8
12(a)	3	3	Blue	0·378	0·282	27·4	0·364	0·284	29·0	0·366	0·284	27·8
7(a)	3	3	Red	0·296	0·307	36·0	0·305	0·317	39·4	0·309	0·319	39·5
6(a)	3	3	Red	0·412	0·363	37·2	0·407	0·361	36·7	0·401	0·361	39·5
5(a)	2	2	—	0·381	0·421	51·1	0·364	0·395	53·2	0·357	0·385	54·5
	GROUP B— LEVEL-DYEING ACID DYES											
13	4–5	4–5	—	0·590	0·320	13·4	0·571	0·320	15·0	0·563	0·320	15·6
14	4–5	4–5	—	—	—	—	—	—	—	—	—	—
15	4	3–4	—	0·525	0·419	33·7	0·508	0·417	39·0	0·498	0·414	39·1
15(a)	4	4	—	0·489	0·414	42·2	0·462	0·406	47·4	0·461	0 406	44·5
16	4	3–4	—	0·557	0·392	28·7	0·548	0·391	29·2	0·534	0·394	30·0
16(a)	4	4	—	0·498	0·398	37·3	0·478	0·388	40·0	0·461	0·383	42·5
19	3–4	3	Blue	0·486	0·267	17·3	0·426	0·250	14·7	0·408	0·240	13·1
20(a)	3–4	3–4	—	0·380	0·431	59·5	0·368	0·415	66·1	0·360	0·403	63·2
17	3	3	—	0·546	0·294	9·8	0·525	0·292	11·4	0·505	0·291	13·4
17(a)	3	3	—	0·443	0·286	20·4	0·414	0·289	26·8	0·400	0·292	29·0
18	3	3	Red	0·190	0·107	5·4	0·223	0·168	7·5	0·226	0·173	7·7
19(a)	3	3	—	0·394	0·264	27·6	0·364	0·267	32·2	0·365	0·263	28·5
20	3	3	—	0·413	0·468	65·5	0·397	0·453	62·8	0·392	0·443	61·2
21	3	2	Green	0·182	0·147	17·7	0·205	0·197	8·0	0·218	0·220	9·2
13(a)	2–3	2–3	—	0·526	0·315	21·0	0·486	0·313	23·1	0·483	0·313	24·0
22	2–3	2–3	Green	—	—	—	—	—	—	—	—	—
22(a)	2–3	2–3	Green	—	—	—	—	—	—	—	—	—
23	2–3	2–3	Red	—	—	—	—	—	—	—	—	—
21(a)	2–3	2–3	—	0·205	0·204	19·7	0·237	0·249	26·5	0·242	0·257	26·0
23(a)	2	2	—	—	—	—	—	—	—	—	—	—
24	2	2	Red	0·184	0·196	9·6	0·195	0·211	9·3	0·205	0·226	10·6
18(a)	2	1–2	Red	0·227	0·182	7·2	0·252	0·231	23·7	0·267	0·255	26·7
24(a)	1–2	2	—	0·209	0·239	22·9	0·245	0·266	26·4	0·255	0·281	29·9
	GROUP C— NEUTRAL-DYEING ACID DYES											
25	4–5	4–5	—	—	—	—	—	—	—	—	—	—
26	4	3–4	—	0·574	0·327	18·1	0·555	0·333	18·9	0·561	0·322	18·6
27	3–4	3–4	Blue	0·472	0·240	12·4	0·432	0·235	11·9	0·403	0·230	11·5
28	3	3	—	0·438	0·483	57·7	0·428	0·468	60·5	0·414	0·463	57·7
29	3	3	Blue	—	—	—	—	—	—	—	—	—
30	3	2–3	Green	0·240	0·218	2·1	0·275	0·258	2·8	0·290	0·275	3·1
31	2–3	2–3	Blue	0·247	0·302	16·8	0·249	0·348	16·9	0·254	0·356	17·3
32	2–3	2–3	Green	0·183	0·167	6·4	0·203	0·204	7·3	0·205	0·208	8·3
33	2–3	2–3	Green	—	—	—	—	—	—	—	—	—
34	2	2	—	—	—	—	—	—	—	—	—	—
34(a)	2	2	—	—	—	—	—	—	—	—	—	—
35	2	2	Blue	0·206	0·316	8·3	0·218	0·311	9·1	0·223	0·314	10·1
30(a)	2	2	Green	0·219	0·213	7·8	0·270	0·261	10·9	0·296	0·276	12·4
32(a)	1–2	2	—	0·213	0·221	15·7	0·230	0·256	23·2	0·252	0·274	27·2
29(a)	1–2	1–2	Blue	—	—	—	—	—	—	—	—	—
33(a)	1–2	1	—	—	—	—	—	—	—	—	—	—
35(a)	1	1	Blue	0·240	0·316	24·0	0·270	0·314	21·1	0·277	0·310	32·9
36	1	1	—	0·177	0·128	7·2	0·219	0·222	14·3	0·231	0·236	16·3
36(a)	1	1	—	0·225	0·212	23·6	0·263	0·275	37·5	0·280	0·295	43·6
26(a)	1	1	—	0·474	0·324	26·9	0·409	0·332	36·5	0·396	0·332	39·8
31(a)	1	1	Blue	0·265	0·362	36·0	0·283	0·332	39·7	0·285	0·329	41·6
27(a)	1	1	Blue	0·391	0·240	22·2	0·358	0·260	28·0	0·341	0·269	30·3
28(a)	1	1	—	0·390	0·432	62·6	0·356	0·388	64·1	0·350	0·377	62·7
	GROUP D— SOLUBLE ACETATE-RAYON DYES											
37	3–4	3–4	—	0·253	0·390	8·4	0·269	0·377	11·7	0·267	0·376	11·9
38	3	3	Red	0·514	0·431	32·3	0·502	0·423	28·8	0·504	0·429	31·2
38(a)	3	3	Red	0·450	0·444	49·5	0·441	0·441	51 0	0·427	0·437	52·4
39	3	2	—	0·330	0·234	4·2	0·336	0·249	9·0	0·344	0·261	9·3
39(a)	2–3	2–3	—	0·313	0·242	13·6	0·319	0·264	21·1	0·321	0·275	25·4
37(a)	1–2	1	—	0·273	0·378	23·2	0·280	0·364	27·8	0·289	0·365	30·4
	GROUP E— DIRECT DYES											
40	2	2	Blue	0·525	0·280	6·2	0·403	0·281	6·2	0·398	0·285	6·2
40(a)	1	1	Blue	0·443	0·272	13·4	0·371	0·308	22·6	0·381	0·297	20·7

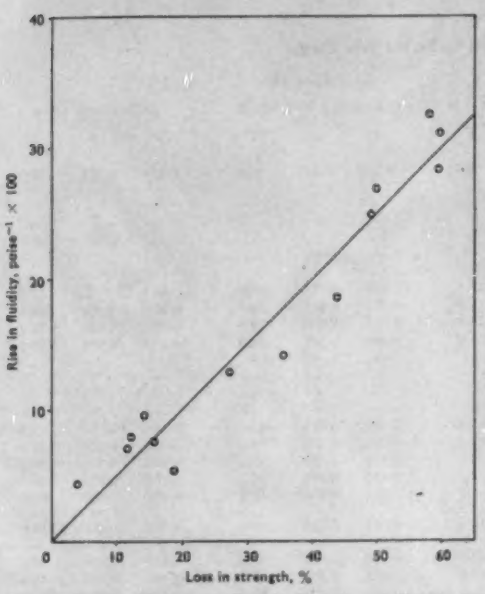

FIG. 2—Relationship between Loss in Strength and Fluidity Rise (Undyed Nylon)

Discussion of Results

1. ALTERATION OF COLOUR (VISUAL ASSESSMENT)

(a) *Effect of Dry Chlorine*

The dyes used consist of three main groups, as follows—

GROUP A—Chrome dyes: twelve heavy and six light dyeings

GROUP B—Level-dyeing acid dyes: twelve heavy and eleven light dyeings

GROUP C—Neutral-dyeing acid dyes: twelve heavy and eleven light dyeings.

Two other types were also included—

GROUP D—Soluble acetate-rayon dyes: three heavy and three light dyeings

GROUP E—Direct dyes: one heavy and one light dyeing.

In Table VI the dyes in each group have been classified into four divisions according to the degree of the colour change which took place during chlorination at 27°C. and clearing with sodium sulphite, the number of dyes falling into each division being recorded.

Examination of ingrain half-hose after chlorination suggests that a change indicated by "3" or higher, viz. divisions III and IV, will be satisfactory for normal use, whilst 2 and 2–3 (division II) are doubtful. On this basis, only Group A

TABLE III

Mean Chlorine Absorptions (%) for Treatment in the Presence of Wool at 50°C.

	Dyeing	
	Heavy	Light
GROUP A		
Free chlorine ...	1·73	1·77
Combined chlorine ...	0·27	0·25
Chlorine on wool ...	1·15	0·98
GROUP B		
Free chlorine ...	1·50	1·61
Combined chlorine ...	0·31	0·36
Chlorine on wool ...	1·10	1·25
GROUP C		
Free chlorine ...	1·87	1·69
Combined chlorine ...	0·37	0·35
Chlorine on wool ...	1·19	1·12

TABLE IV

Colour Assessment of Fastness to Wet Chlorination of Dyes on Nylon and Wool

Dye (ICI)	Depth (%)	Wool*	Fastness on Nylon		Change in Hue of Nylon towards
			Chlorinated with Wool	Chlorinated Alone	
Solochrome Black 6 BNS	10·0	4	4	3–4	Green
Solochrome Red ERS	1·0	4	4	3–4	Blue
Solochrome Red ERS	0·2	—	3	3	Blue
Naphthalene Orange GS	1·5	4–5	4–5	4	—
Naphthalene Orange GS	0·3	—	4–5	4–5	—
Lissamine Fast Violet 2 BS ...	1·5	3	3–4	3–4	Red
Lissamine Fast Violet 2 BS ...	0·3	—	3–4	2	Red
Solway Blue BNS	1·25	3	4	4–5	—
Solway Blue BNS	0·25	—	3–4	3	—
Solway Green GNS	1·0	4	4	3–4	—
Solway Green GNS	0·2	—	3	2–3	—
Carbolan Green GS	1·5	4–5	4	4–5	Blue
Carbolan Green GS	0·3	—	3	2–3	Blue
Coomassie Brilliant Blue RS ...	2·0	3	3–4	3	Red
Coomassie Brilliant Blue RS ...	0·4	—	3–4	1–2	Red
Solacet Fast Orange 2 GKS ...	1·5	—	3–4	3	Red
Solacet Fast Orange 2 GKS ...	0·3	—	4	3	Red
Solacet Fast Red 5 BGS	1·25	—	4	4	—
Solacet Fast Red 5 BGS	0·25	—	3–4	2–3	—
Chlorazol Fast Red FS	1·5	—	3–4	3	Blue

*These figures have been taken from the dye-manufacturer's pattern cards and refer to medium depths except for Solochrome Black 6 BNS (8%).

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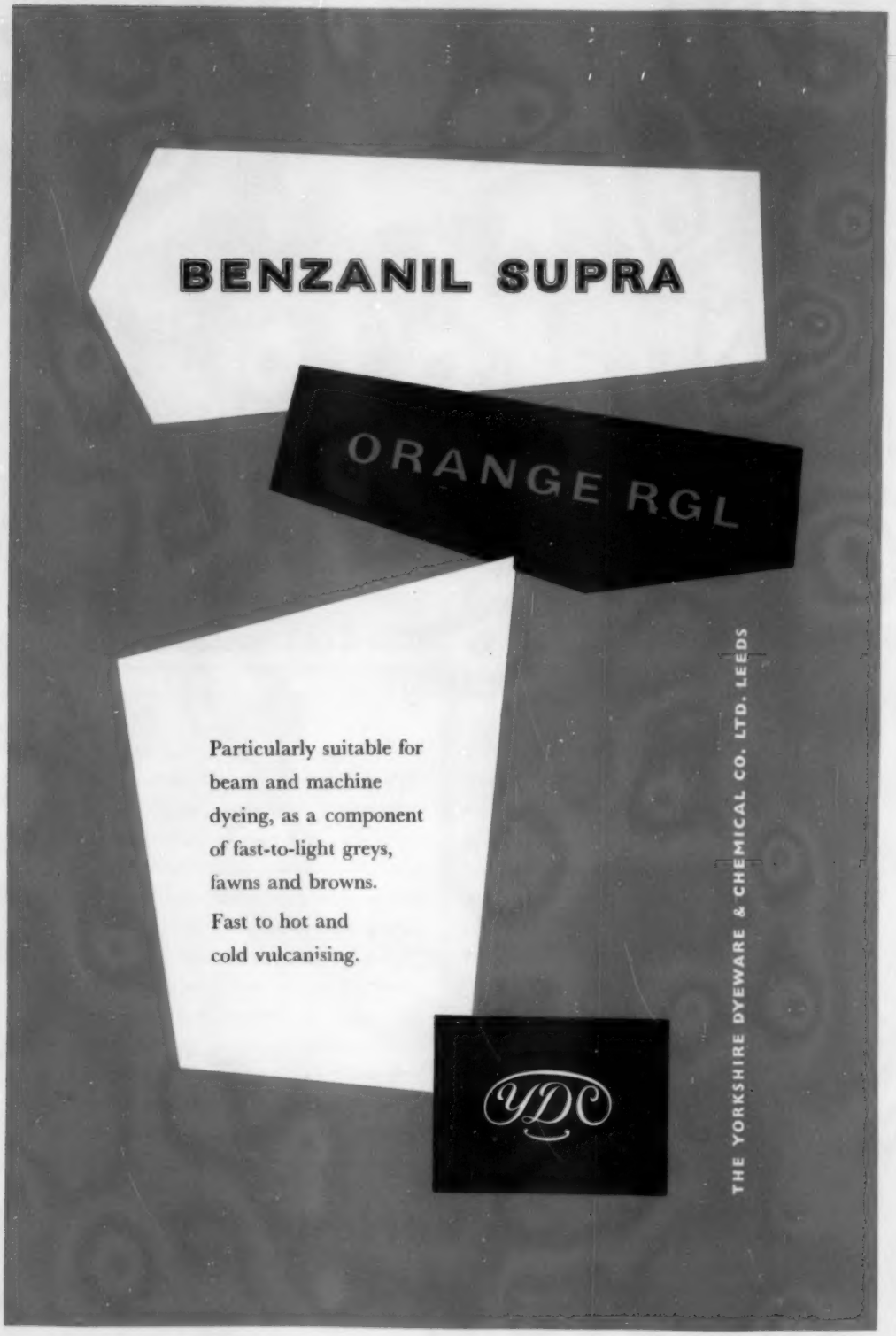
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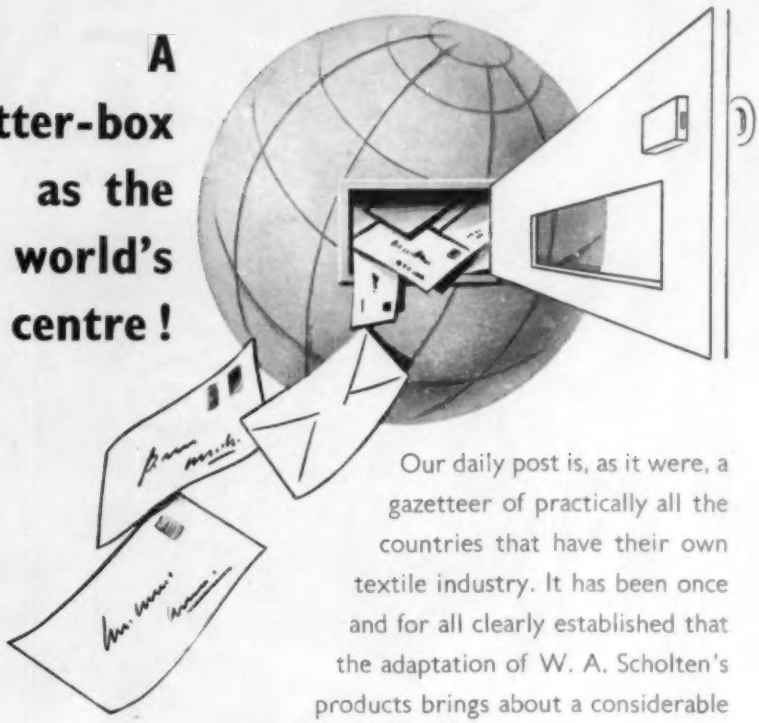

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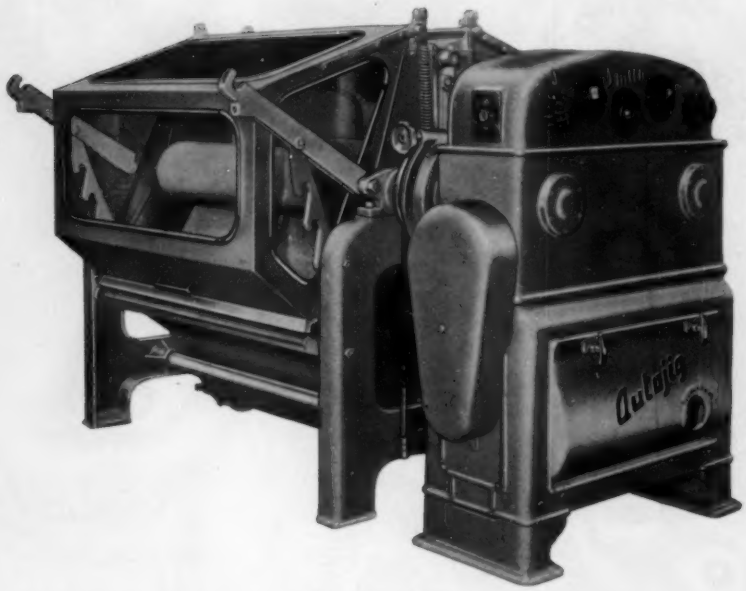

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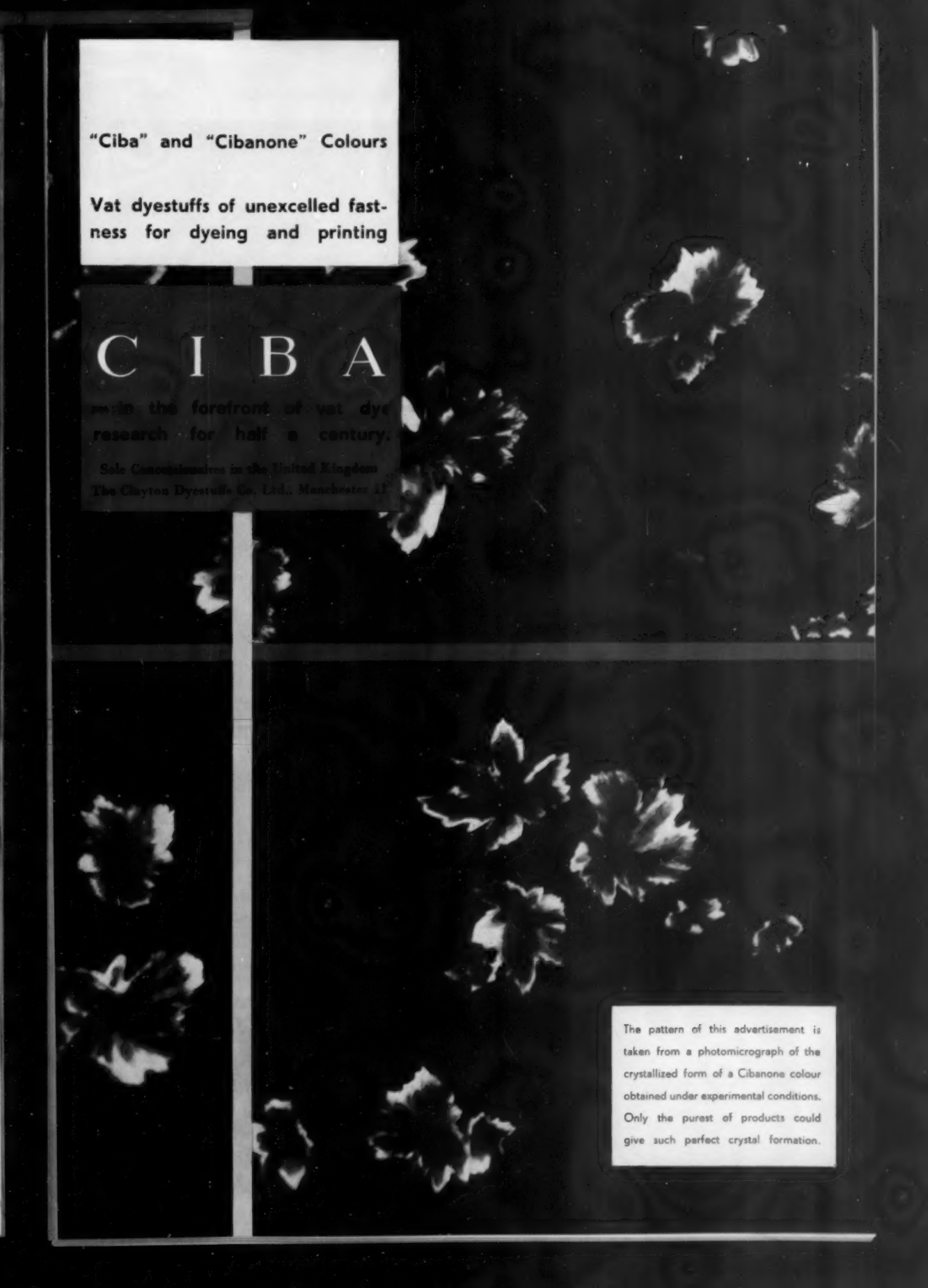
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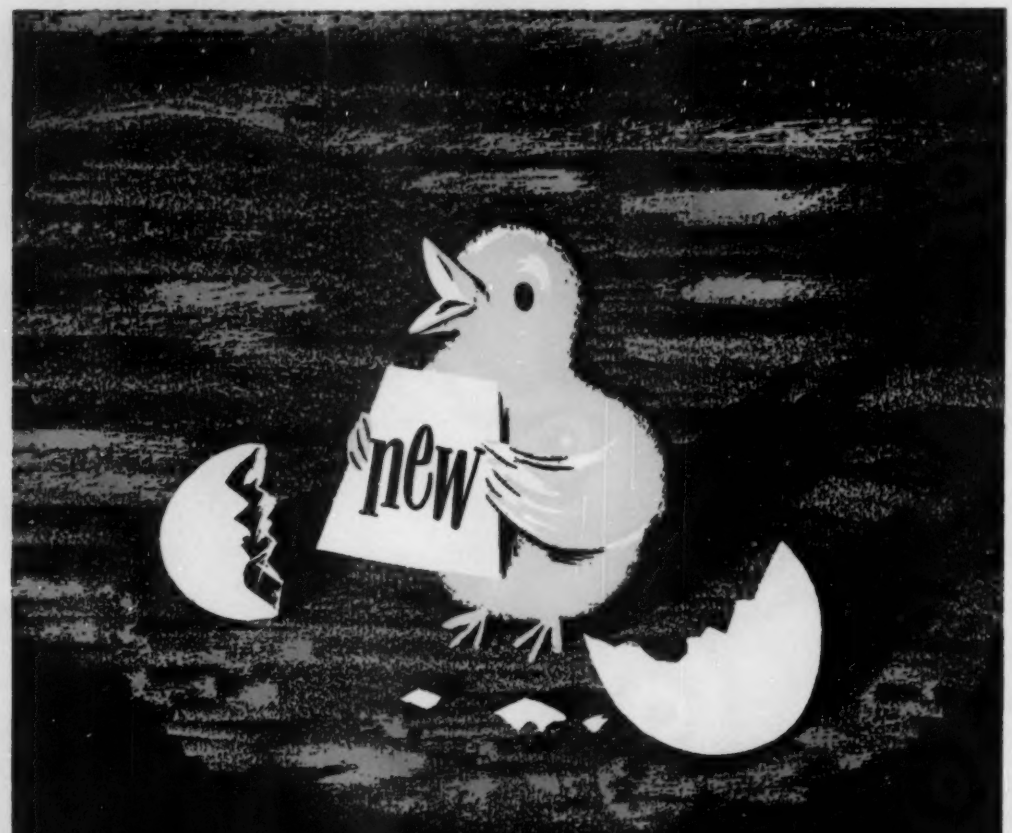

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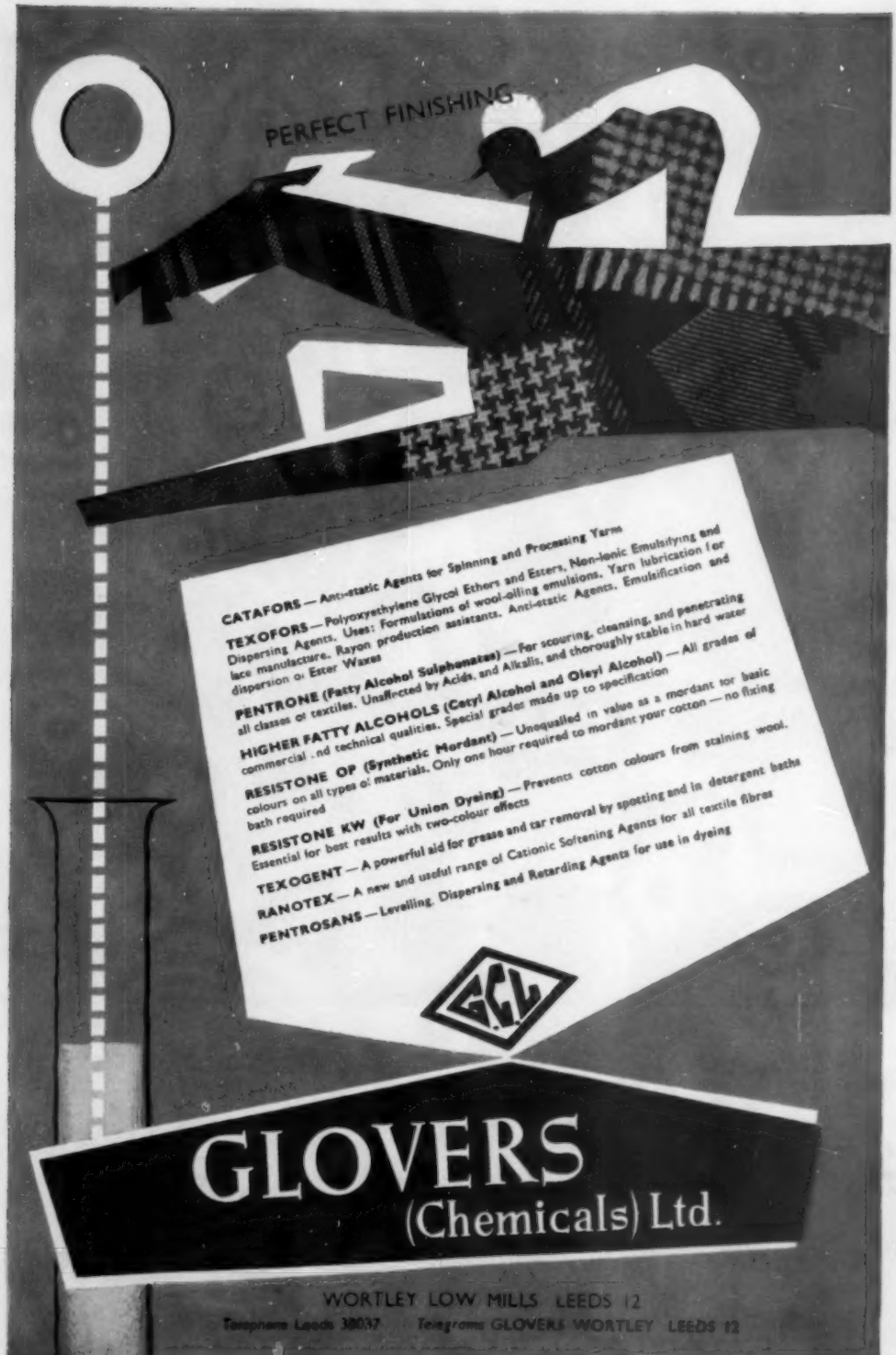
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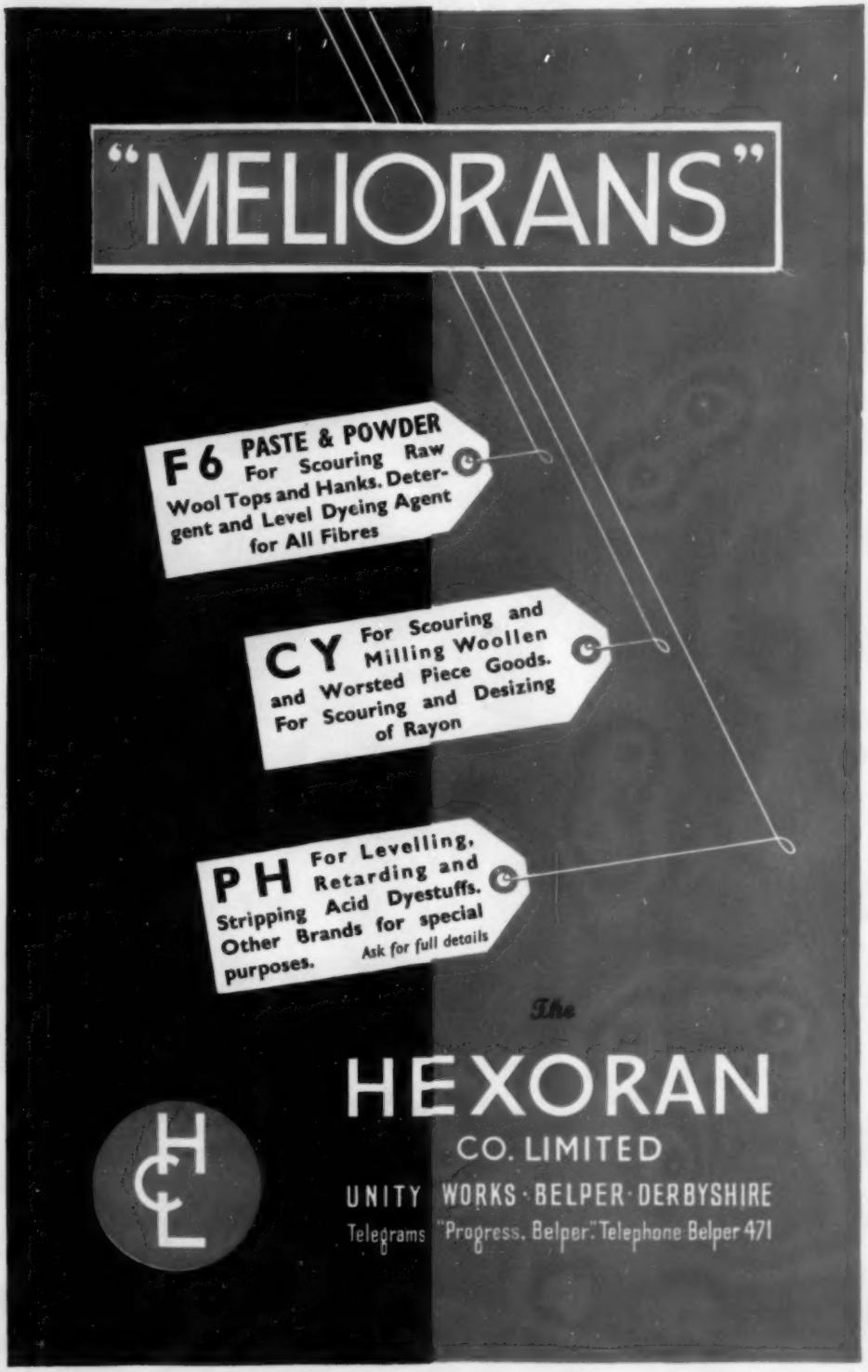
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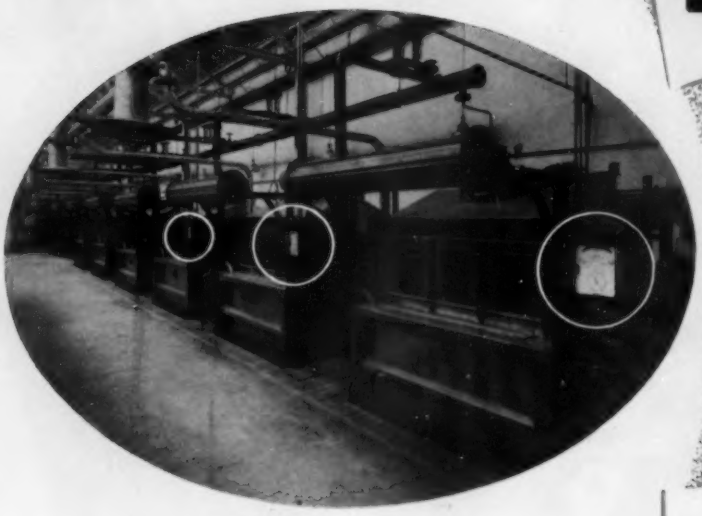

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(chrome) dyes are wholly satisfactory, whilst the lighter dyeings of Group C (neutral-dyeing acid dyes) are useless. All the others will need individual examination to determine their suitability. (Groups D and E must be neglected in this discussion, as an insufficient number of dyes were tested.) No substantial differences were found when the conditions of chlorination and neutralisation were altered, e.g. chlorination at 50°C. and neutralisation by sodium bisulphite.

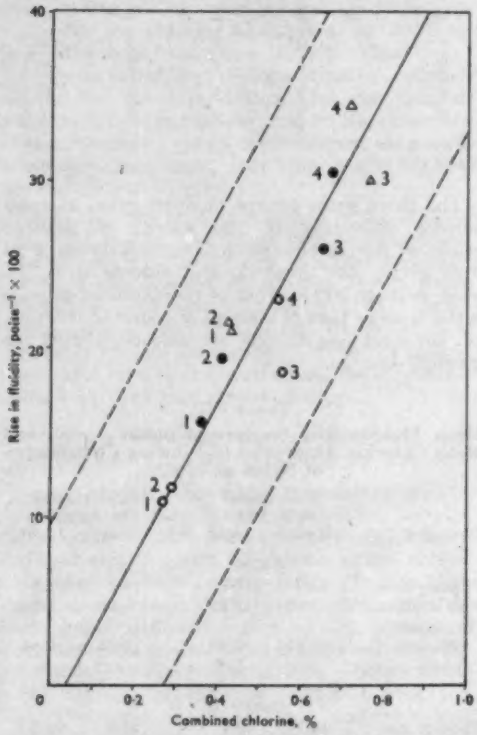
Rise in fluidity, poise^{-1} × 100
Combined chlorine, %
A ○ Chrome dyes
B ● Level-dyeing acid dyes
C △ Neutral-dyeing acid dyes
1 Heavy dyeing with wool
2 Light dyeing with wool
3 Heavy dyeing of nylon alone
4 Light dyeing of nylon alone

FIG. 3 — Relationship between Combined Chlorine and Fluidity Rise (Dyed Nylon)

TABLE V

Colour Fastness Assessment of Samples treated with Hydrogen Peroxide

Dye (ICI)	Dyeing Heavy	Dyeing Light
Solochromate Fast Orange 2 RS ...	4	—
Solochrome Red ERS	3–4	1
Naphthalene Orange GS	3–4	—
Solway Ultra Blue BS	3–4	—
Coomassie Red PGS ...	...4, to yellow	4–5
Coomassie Navy Blue 2 RNS ...	4–5	—
Carbolan Brilliant Green 5 GS ...	4–5	4–5
Coomassie Brilliant Blue RS ...	—*	—*

*This sample changed in hue from bright blue to pale green, with almost complete stripping.

(b) *Effect of Wet Chlorination*

With wet chlorination, a higher fastness is generally obtained; for all the forty samples examined, the mean difference is less than 1 unit. Eight samples— Coomassie Brilliant Blue RS (three), Carbolan Green GS (three), Solway Blue BNS (one), and Solacet Fast Red 5 BGS (one)— show differences of 2 or 2½ units. In one case— Solochrome Black 6 BNS— the dye was rather less fast to wet chlorine than to dry, a change of hue also occurring.

It is of interest to note that in a recent publication [9] many of the dyes listed in Tables I and II have been examined for fastness to wet chlorination. Level-dyeing acid, neutral-dyeing acid, soluble acetate-rayon, and direct dyes all show a slightly greater fastness to wet than to dry chlorine. Only two chrome dyes out of twelve show this result: all the others are faster to dry chlorine than to wet. The figures given in Table IV for fastness to wet chlorine are in close agreement with those given for the same dyes in the above publication [9].

(c) *Effect of Hydrogen Peroxide*

Of the twelve dyes examined, only three showed a greater fading by hydrogen peroxide than by dry chlorine, Coomassie Brilliant Blue RS being completely changed in hue to green. The remaining nine dyes were only slightly affected by the treatment.

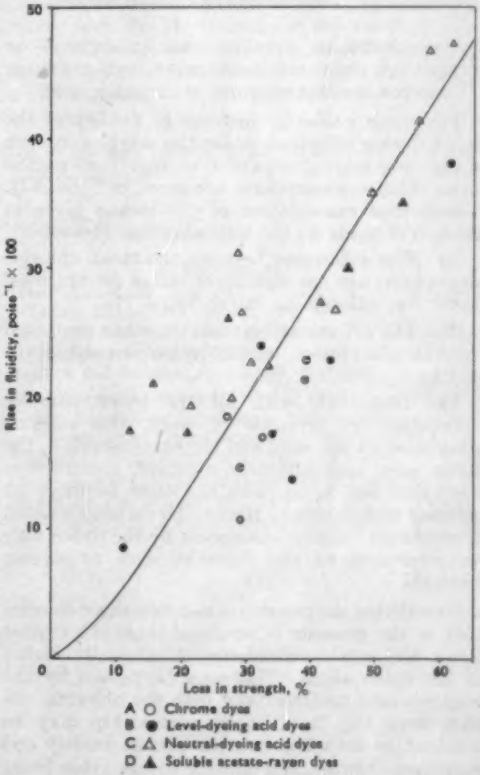
Rise in fluidity, poise^{-1} × 100
Loss in strength, %
A ○ Chrome dyes
B ● Level-dyeing acid dyes
C △ Neutral-dyeing acid dyes
D ▲ Soluble acetate-rayon dyes

FIG. 4 — Relationship between Loss in Strength and Fluidity Rise (Dyed Nylon)

TABLE VI

Division of Dye Groups according to Change of Colour

Dye Group and Dyeing*	Total No. of Dyes	No. of Dyes in Each Division							
		Chlorinated with Wool				Chlorinated Alone			
		I	II	III	IV	V	VI	VII	VIII
		<2	2, 2–3	3, 3–4	4, >4	<2	2, 2–3	3, 3–4	4, >4
A—Heavy ...	12	0	0	5	7	0	0	5	7
A—Light ...	6	0	1	4	1	0	1	4	1
B—Heavy ...	12	0	3	5	4	0	4	6	2
B—Light ...	11	1	4	4	2	1	4	4	2
C—Heavy ...	12	1	5	4	2	1	6	4	1
C—Light ...	11	9	2	0	0	9	2	0	0
D—Heavy ...	3	0	0	3	0	0	1	2	0
D—Light ...	3	1	1	1	0	1	1	1	0
E—Heavy ...	1	0	1	0	0	0	1	0	0
E—Light ...	1	1	0	0	0	1	0	0	0

*Heavy dyeing: 0·75–10% dye
Light dyeing: 0·15–0·45% dye.

2. ALTERATION OF COLOUR BY DRY CHLORINE (QUANTITATIVE METHOD)

Examination of chromaticity diagrams drawn from the data in Table II confirms the changes in colour obtained by visual assessment for the particular dyes examined. Slight changes in hue are indicated for three other dyes, the change being too slight to be estimated visually. These dyes are 16 (change towards yellow), 26 (change towards green), and 40 (change towards blue).

3. INFLUENCE OF TYPE OF DYE OR METHOD OF DYEING ON CHLORINE ABSORPTION AND FLUIDITY CHANGE OF NYLON DURING CHLORINATION

The mean values of increase in fluidity of the nylon during chlorination for the samples in each of the three main groups of dyes, together with the mean chlorine absorptions, are given in Table VII.

Statistical examination of the results given in Table VII leads to the following conclusions—

(*a*) The differences between the mean chlorine absorptions are not significant either for the wool or for free chlorine on the nylon.

(*b*) The differences between the mean combined chlorine absorptions on the nylon are significant at 1%.

The fact that, with all the nylon samples chlorinated in presence of wool, the chlorine absorption by the wool and the free chlorine in the nylon were approximately constant, implies that the nylon had in all cases the same facilities for reacting with chlorine. Hence, the variations noted in combined chlorine absorption by the nylon may be attributed to the different dyes or dyeing methods.

Considering the two sets of samples, those chlorinated in the presence of wool and those chlorinated alone, the combined chlorine is markedly higher in the nylon alone. This may be caused by the wool reacting preferentially with the chlorine. As seen from Fig. 3, a linear relationship may be assumed to exist between the rise in fluidity and combined chlorine, less damage to the nylon being associated with a lower combined chlorine content.

The three main groups of dyed nylon samples behave differently in the extent of damage produced during chlorination, neutral-dyeing acid dyes giving the greatest, and chrome dyes the least, damage. This order of resistance to damage is the same as that of fastness of colour to chlorine, i.e. the least fast colours are associated with the greatest damage.

TABLE VII

Mean Fluidity Rise (reciprocal poises × 100) and Mean Chlorine Absorption (%) during Chlorination of Nylon at 27°C.

Dyeing ...	Chlorinated with Wool		Chlorinated Alone	
	Heavy	Light	Heavy	Light
GROUP A				
Fluidity rise ...	10·9	11·8	18·5	22·8
Free chlorine ...	1·00	1·20	1·36	1·38
Combined chlorine ...	0·27	0·29	0·55	0·54
Chlorine on wool	0·99	1·02	—	—
GROUP B				
Fluidity rise ...	15·7	19·4	25·9	30·5
Free chlorine ...	1·07	1·13	1·49	1·50
Combined chlorine ...	0·36	0·41	0·65	0·67
Chlorine on wool	0·92	0·99	—	—
GROUP C				
Fluidity rise ...	21·2	21·6	30·2	34·5
Free chlorine ...	1·15	1·27	1·47	1·60
Combined chlorine ...	0·43	0·42	0·76	0·71
Chlorine on wool	1·02	0·99	—	—

Considering the individual dyes in each group, only four give results very different from the others; these are—

GROUP A—Solochrome Black WDFA (10% concentration) gives a low fluidity rise with average chlorine absorption and excellent colour fastness.

GROUP B—Naphthalene Red JS (1% concentration) gives a high fluidity rise with average chlorine absorption and reasonable colour fastness.

GROUP C—Coomassie Red PGS (1·75% concentration) gives a high fluidity rise with low chlorine absorption and good colour fastness in the heavy dyeing and poor fastness in the light dyeing. Coomassie Green WN (10% concentration) gives a low fluidity rise with low chlorine absorption and good colour fastness.

All the other dyes reflect the characteristics of their particular group.

Although it has been stated earlier (p. 241) that variations in the experimental methods have no effect on the colour changes taking place, the results for the chlorine absorptions at 50°C. do show differences from those at 27°C. These are—(*a*) a greater variability; (*b*) the amount of combined chlorine for the level-dyeing and the neutral-dyeing acid dyes is reduced; and (*c*) the difference between the mean values for combined chlorine is statistically significant only for neutral-dyeing acid and chrome dyes (at 1%).

The fluidity of a random selection of the samples chlorinated at 50°C. was determined, and in general a slightly higher fluidity rise was obtained than that from the samples chlorinated at the lower temperature. Compared with undyed nylon chlorinated at 27° and 50°C., the dyed samples show a tendency for less damage to occur in the case of chrome and level-dyeing acid dyes, whilst neutral-dyeing acid dyes give greater damage.

4. RELATION BETWEEN RISE IN FLUIDITY AND AMOUNT OF CHLORINE ABSORBED BY NYLON

Since it has been shown that the only variable in the chlorine absorbed by the dyed nylon samples when treated at 27°C. is that represented by the combined chlorine, any differences in the rise in fluidity may be related to this factor. In Fig. 3 the fluidity rise has been plotted against the combined chlorine, and although a considerable amount of scatter occurs, a general trend is observed, showing that the fluidity rise is approximately proportional to the amount of chlorine combined with the nylon. Comparison between Fig. 1 (undyed nylon) and Fig. 3 is not possible for two reasons—(*a*) the viscosity measurements of the two series were made at different temperatures, and (*b*) the temperature of chlorination for the undyed samples was varied.

5. RELATION BETWEEN RISE IN FLUIDITY AND LOSS IN STRENGTH AT BREAK OF NYLON

In Fig. 2 and 4 the rise in fluidity of nylon is plotted against the percentage loss in strength at break. In both cases, undyed and dyed, the relationship is seen to be linear. Boulton and Jackson[3] showed that a linear relationship exists between loss in strength at break and the rise in fluidity for exposure to ultraviolet radiation and for acid degradation. In the present experiments, the attack on the nylon is probably caused in two ways, viz. oxidation by chlorine and hydrolysis by hydrochloric acid, and assuming that these two modes of attack are independent it might be expected that a combination of them would be linear. This has been confirmed.

6. RELATION BETWEEN STRENGTH AND EXTENSION AT BREAK OF DYED NYLON AFTER DRY CHLORINATION

For the samples tested, both dyed and undyed, a linear relationship was obtained between strength and percentage extension at break, a reduction in strength from 60 oz. to 40 oz. corresponding to a loss of 7½% in extension.

* * *

The authors' thanks are due to Messrs. Imperial Chemical Industries Ltd. for the gift of the samples of dyed nylon yarns and comparative assessment of colour fastness on certain samples; to Dr. F. O. Howitt, Chief Chemist to the Association, for helpful advice; to Mr. F. L. Warburton for advice and assistance in the determination and interpretation of the colorimetric data; to Miss A. Greaves and Mr. N. Warburton for assistance in the experimental work; and to the Director of Research and the Council of the Wool Industries Research Association for permission to publish this paper.

WOOL INDUSTRIES RESEARCH ASSOCIATION
"TORRIDON"
LEEDS 6

(*Received 16th October 1954*)

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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Heat of Oxidation of Sodium Formaldehyde-sulphoxylate

The problem of explaining the temperature rise in an ager or steamer during the steaming of vat dye prints has led us to make a direct determination of the heat of oxidation of sodium formaldehyde-sulphoxylate (Formosul), since the exothermal heat of hydration of an alkali-metal carbonate did not appear to be wholly responsible for the magnitude of the temperature rise experienced. The reaction on which thermal measurements were made was the quantitative oxidation of Formosul by iodine. From the measured heat of this reaction it is possible, with the aid of standard heats of formation, to calculate the heat of combination of Formosul with gaseous oxygen.

The thermal measurements were carried out at 25°C. in a calorimeter which has been fully described elsewhere[1]. For use with iodine an all-glass pipette replaced that of metal, glass, and rubber normally employed; otherwise the method of operation was as previously described. 100 ml. of a standard 0·1 N. solution of iodine in potassium iodide solution was added from the mixing pipette to 900 ml. of Formosul solution in the body of the calorimeter. The heat change was measured, and the excess iodine subsequently determined by titration with sodium thiosulphate. A small correction for the heat of dilution of the iodine solution on mixing was made from the result of a separate experiment in which 100 ml. of 0·1 N. iodine was added to 900 ml. of water. Any heat change due to the small dilution of the Formosul was ignored. Three experiments were done in this way, giving values for the heat of reaction of −37·6, −37·3, −37·3 kcal. per mole of Formosul.

Thus for the reaction—

$$NaHSO_2,CH_2O + 2I_3^- + 2H_2O\,(l) \rightarrow CH_2O + NaHSO_4 + 4H^+ + 6I^- \qquad (i)$$

the heat of reaction is $\Delta H_1 = -37{\cdot}4$ kcal. per mole of Formosul. Then if the reaction of Formosul with gaseous oxygen is assumed to proceed according to the equation—

$$NaHSO_2,CH_2O + O_2(g) \rightarrow CH_2O + NaHSO_4 \qquad (ii)$$

the heat of this reaction (ΔH_2) may be calculated as follows—

Subtraction of equation (ii) from equation (i) gives—

$$2I_3^- + 2H_2O(l) \rightarrow O_2(g) + 4H^+ + 6I^- \qquad (iii)$$

to which hypothetical reaction a value $\Delta H_3 = \Delta H_1 - \Delta H_2$ may be ascribed. ΔH_3 may be calculated as the difference between the standard heats of formation of the products and reactants of equation (iii), all of which are given in National Bureau of Standards tables[2]. This procedure gives $\Delta H_3 = +81{\cdot}2$ kcal. per mole of O_2. Therefore $\Delta H_2 = \Delta H_1 - \Delta H_3 = -37{\cdot}4 - 81{\cdot}2 = -118{\cdot}6$ kcal. per mole of Formosul.

This result indicates that air oxidation of Formosul produces heat to the extent of about 1000 calories per gram of Formosul oxidised, and the reaction is so strongly exothermal that oxidation by this mechanism of only about one-tenth of the Formosul in a typical print-paste formulation will produce temperature rises of the order of magnitude of those observed in a steamer. Thus there is little doubt that oxidation of Formosul, whether by oxygen itself (i.e. residual air in the steamer) or by a vat dye, is a primary cause of the observed effect.

A. N. Derbyshire
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23rd March 1955

[1] Derbyshire and Marshall, in "The Physical Chemistry of Dyeing and Tanning" (*Discussions Faraday Soc.*, (16), 140 (1954)).

[2] "Selected Values of Chemical Thermodynamic Properties", *National Bur. Stand. Circular* No. 500 (1952).

Notes

Postal Ballot 1955

The numbers of votes cast for the different candidates in the postal ballot for the election of Vice-presidents and Ordinary Members of Council (see p. 214) were as follows—

Vice-presidents

Mr. H. à Brassard	308
Mr. A. Breare	278
Mr. W. A. Edwards	354
Mr. R. J. Hannay	468
Mr. G. G. Hopkinson	382
Mr. G. S. J. White	397

Ordinary Members of Council

Mr. A. S. Cluley	247
Dr. A. Datyner	144
Mr. J. M. Goodall	351
Dr. R. H. Peters	218
Dr. G. R. Ramage	168
Dr. W. Shaw	183
Mr. A. Thomson	312
Mr. R. Thornton	214
Dr. T. Vickerstaff	401

A total of 634 ballot papers were received, of which 12 were spoilt and 10 were disallowed because they were received late. The 612 valid papers thus contained a total of 4425 votes.

Extraordinary General Meeting Honorary Secretary of the Society

At an Extraordinary General Meeting of the Society held at the Victoria Hotel, Bradford, on Wednesday, 27th April 1955, Mr. Lascelles Morton Wood was elected Honorary Secretary of the Society, in accordance with the nomination of Council (see p. 214).

The election of Mr. L. Morton Wood was proposed by Mr. Fred Smith (*President of the Society*, in the chair), who spoke of Mr. Wood as a well known business gentleman of Bradford; seconded by Mr. H. Jennison (*Honorary Treasurer*), who said that in Mr. Wood the Society was very fortunate in having a businessman whose ability would be of great value to the Society; and carried with acclamation.

The President extended a warm welcome to Mr. L. Morton Wood and paid high tribute to Mr. J. G. Hopkinson, the retiring Honorary Secretary.

Mr. L. Morton Wood, the only son of the late Norman Wood, is managing director of Norman Wood & Son Ltd., Crown Dyeworks, Bradford, and Vice-president of the Dyers and Finishers Association. He was educated at Leys School, Cambridge, and also attended Bradford Technical College. During the Second World War he served in the Royal Artillery, achieving the rank of Major.

Meetings of Council and Committees April

Council— 27th

Chairmen and Honorary Secretaries— 2nd

Finance— 27th

Publications— 19th

Colour Index Editorial Panel— 15th

Textbooks Subcommittee— 14th

Diplomas Executive Subcommittee— 19th

Perkin Executive Subcommittee— 18th

Deaths

We regret to report the loss by death of Mr. J. R. Berwick and Mr. T. N. T. Graham.

Annual Meeting of Section Officers

The Annual Meeting of Chairmen and Honorary Secretaries of Sections was held at the Queens Hotel, Leeds, on Saturday, 2nd April 1955, under the chairmanship of Mr. H. Jennison (Honorary Treasurer of the Society). The topics discussed included the membership card, distribution of the brochure and the recruitment of new members, and the circulation of section notices.

Huddersfield Technical College School of Dyeing 1841–1944

The school of dyeing at Huddersfield Technical College is one of the oldest, perhaps the oldest, in the country. Mr. H. Wilkinson, a former head of the school, has written a history of it, illustrated by photographs of former members of the staff and of the laboratories. Three copies have been prepared, one of which has been presented to the Society by Mr. Wilkinson. This copy was displayed at the Annual General Meeting of the Huddersfield Section on 15th March 1955, when Mr. C. O. Clark, Chairman of the Society's Committee for the Preservation of Historical Records, announced the gift and expressed the deep thanks of the Society to Mr. Wilkinson. It was decided to send a letter of appreciation to Mr. Wilkinson from the meeting, which included many of his former students.

C. O. C.

Twentieth Anniversary of the CATCC

The twentieth anniversary of the formation of the Ontario Section of the Canadian Association of Textile Colourists and Chemists was celebrated at Hamilton on 5th February 1955. The inauguration of the Ontario Section in Hamilton in February 1935 was followed by that of the Quebec Section in Montreal in March 1936. These led to the formation in 1937 of the Textile Society of Canada (with Western and Eastern Divisions) to cover the manufacturing aspects of textiles. The need for a co-ordinating body was felt, and in February 1939 the Textile Technical Federation of Canada was established. This comprises also the Cornwall and District and the Ottawa Valley Textile Associations, formed after the Second World War.

AATCC Convention 1955

This will be held during September 22nd–24th at Chalfonte-Haddon Hall, Atlantic City N.J. The technical programme will emphasise mill problems of the dyer and finisher. Further information is available from the AATCC, Lowell Technological Institute, Lowell, Mass., U.S.A.

Society of Chemical Industry Medal

Professor Sir Eric Rideal, M.B.E., F.R.S., will receive the Medal of the Society of Chemical Industry during the A.G.M. of the S.C.I. to be held in Birmingham in July 1955. He will deliver an address on "Colloid Science".

Liability of Scientific Societies to Payment of Rates

In a recent case, the Court of Appeal held (*Dyer*, **113**, 133 (21st Jan. 1955)) that the Institute of Fuel was not entitled under the Scientific Societies Act 1843 to exemption from rating, on the ground that its objects included "(*d*) to uphold the status of members of the Institute by holding or prescribing examinations for candidates for election and by requiring standards of knowledge and experience which can be approved." In the view of Lord Justice Jenkins, who dissented, the objects aimed at in paragraph (*d*) were the promotion of the science of fuel technology, and any advantages accruing to members of the Institute were purely ancillary.

Centenaries in 1955

This year is the centenary of the birth of C. F. Cross (1855–1935), who in 1892 obtained viscose from cellulose, and thus laid the foundations of the rayon industry.

Another interesting centenary is that of the death of the chemist C. B. Mansfield (1819–1855), who discovered a method of extracting benzene from coal-tar. He died of burns received when a naphtha still overflowed during the preparation of samples of benzene for the Paris Exhibition.

Consumption of Sulphuric Acid

According to statistics prepared by the National Sulphuric Acid Association Ltd. (*Chem. and Ind.*, 159 (12th Feb. 1955)) the total consumption of sulphuric acid in the United Kingdom during the year 1954 was 2,047,748 tons of 100% H_2SO_4. This total included 83,759 tons for dyes and intermediates, 248,161 tons for rayon and transparent paper, and 20,552 for textile uses.

Japanese Export of Dyes in 1954

According to the Japanese Finance Ministry, Japan exported 1808 metric tons of dyes in 1954, a drop of 15% on the 1953 export of 2140 metric tons. In classes of dyes the 1954 exports were (in metric tons)—acid 65; basic 113; direct 242; indigo—natural < 500 kg., synthetic 3 (354 in 1953); lakes 40; mordant 28; solvent nil; sulphur 1104; vat 39; miscellaneous 170. C. O. C.

Cadmium Pigments

According to the British Bureau of Non-ferrous Metals, 172 tons of cadmium metal out of a total consumption of 731 tons was used in 1954 to make pigments; this compares with 130 tons out of 622 tons in 1953. C. O. C.

Journal für praktische Chemie

The publishing house of Johann Ambrosius Barth of Leipzig announces that it is to resume publication of the above journal. Three volumes each consisting of six issues will appear each year, the price of each volume being DM 24.00. In effect the journal will be the organ of the Chemische Gesellschaft der Deutschen Demokratischen Republik.

Research on Textiles in Canada

According to the *Thirty-seventh Annual Report of the National Research Council of Canada, 1953–54* (N.R.C. No. 3286. Ottawa 1954), the Division of Applied Chemistry has collaborated with the Canadian Research Institute of Launderers and Cleaners in studying the sorption of surface-active agents by textile fibres, factors affecting the soiling characteristics of textile fibres, the microbiological and ultraviolet breakdown of cellulosic fibres, and methods of measuring damage in the newer synthetic fibres. Work has been started on the oxidation of polyvinyl alcohol with Fenton's reagent.

Man-made Fibre Capacity in the U.S.A.

According to the *Rayon Organon*, it is estimated that the current capacity for the production of man-made fibres in the U.S.A. is $2{\cdot}2 \times 10^9$ lb. a year. This is expected to increase to $2{\cdot}3 \times 10^9$ lb. by the end of 1956.

Dralon Polyacrylonitrile Fibre

Dralon (FBy) polyacrylonitrile fibre, which has recently been placed on the market, has wool-like properties and is of low specific gravity. A 30–40% addition to wool is claimed to prevent felting of the latter.

Physical Chemistry of Enzymes

A General Discussion on this subject will be held by the Faraday Society at Oxford during 10–12th August 1955.

ISO Meeting on Safety Colours

The first meeting of ISO Technical Committee ISO/TC 80, on "Safety Colours", was held at the Hague during 5–9th October 1954. At the suggestion of the International Labour Office it considered the setting up of an international standard for safety colours, and appointed a subcommittee to define scientifically three such colours, e.g. green, red, and orange–yellow.

Award of Chardonnet Medal to Dr. H. A. Thomas

The Hilaire de Chardonnet medal has been awarded to Dr. H. A. Thomas by the French Producers of Man-made Fibres in recognition of his services to the International Rayon and Synthetic Fibres Committee. This medal commemorates the initiation of the rayon industry in 1891 by Chardonnet (1839–1924) in Besançon.

New Books and Publications

Food Standards Committee Report on Colouring Matters

Ministry of Food. Pp. 27. London: H.M.S.O. 1954. Price, 1s. 0d.

The Food Standards Committee of the Ministry of Food has adopted the report of its Preservatives Subcommittee, with its recommendation that the Public Health (Preservatives, etc. in Food) Regulations should be amended so as to permit the use in foods of specific dyes only. The Food Standards Committee has thus endorsed the recommendation of the Departmental Committee appointed by the Minister of Health in 1923, a recommendation not implemented by the Government of the day, which preferred to prohibit the use of certain dyes only, which were known to be harmful. The recommendations of the Food Standards Committee specify a "List of Permitted Colours", reproduced as Appendix III to the Report, comprising 13 colouring matters of natural origin (amongst which is placed Carbon Black) and 32 synthetic dyes.

The Report records the arguments presented to the Preservatives Subcommittee and the conclusions reached on the questions whether the addition of colouring to food is necessary or desirable, whether the addition of colouring to particular foods or classes of foods should be prohibited, whether the system of control should be through a prohibited list or a permitted list and, accepting the latter alternative, the dyes to be permitted in foods and the specifications of purity to which they should conform. Considerations and conclusions concerning regulation of the amounts of colouring to be used, labelling, control of the sale of food dyes, and provisions to deal with imported foods are also recorded.

There are three appendixes, the third of which gives the names, chemical constitutions, and, where they exist, *Colour Index* numbers of the recommended list of permitted dyes. Appendix I, a valuable survey of "coal-tar colours" with special reference to the pharmacological aspect, emphasises carcinogenicity, listing some thirty selected references worthy of special study; while Appendix II presents the conclusions and advice of a panel of the medical members of the Subcommittee, who studied the pharmacological evidence.

A characteristic activity of our Society at the present time is the drawing up of detailed instructions for the carrying out of agreed tests by which the textile-dyeing properties of dyes shall be assessed in relation to the actual conditions of their use, and members of the Society will therefore be the first to appreciate the difficulties faced by the Subcommittee through the absence of any formulation of agreed biological tests which could form the basis of judgment of the suitability of dyes for use in foods. The medical panel indeed found itself, in the absence of conclusive evidence, unable to recommend any dye unreservedly as safe; while 35 dyes, out of 79 reviewed, were placed in the category of those which have been shown, or are suspected, to have harmful effects. The recommended "Permitted List" is therefore a tentative one, which, the medical panel considers, should be reviewed after an interval of three to five years, during which agreement on biological tests should be reached and they should be applied to a thorough evaluation of all dyes now on the "Permitted List" or any dye subsequently proposed for inclusion.

Implementation of this report should, so far as colouring matters are concerned, allay much of the alarm frequently expressed lest the public health should be endangered by an increasing use in foods of synthetic chemicals without adequate proof that they are free from harmful effects.

M. LAPWORTH

Veredler-Jahrbuch
Deutscher Färberkalender 1955
(Volume 59)

Edited by O. Mecheels. Pp. v + 367 + 61. Munich: Franz Eder Verlag. Price, $3.00.

The high standard of previous issues has been well maintained in the current publication. Twenty-seven articles occupy by far the largest portion of the book, covering recent developments in the dyeing and processing of textiles over a very wide field with, as would be expected, emphasis on the newer synthetic fibres and their admixture with natural fibres such as wool.

Notes on the dyeing of these newer fibres occupy considerable space, but articles such as those dealing with the dyeing of hats and on the use of levelling agents in wool dyeing show that new developments in the dyeing of older fibres have not been overlooked.

The reader interested in a particular type of dye will find separate papers dealing with acid, azoic, direct, sulphur, vat, and solubilised vat dyes, while the printer will find the articles on the mechanisation of screen printing, the steaming of vat dyes, and the use of azoic dyes in calico printing of special interest.

An interesting development is the inclusion of two articles, on the new generation of specialist and professional men in the textile-processing industries and on the organisation of the works laboratory in the same field. Useful lists of German synthetic fibres, giving their makers, deniers, and staple fibre lengths, and tables for the identification of detergents and cleansing agents are included, and the increased number of pages listing new dyes and auxiliary products put on the market during 1954 by German and Swiss firms testifies to the activity of the dyemakers of these two countries.

The wide scope of the articles enables readers to keep in touch with the latest developments in dyeing and textile processing, and the very large amount of information so clearly set out makes the volume one which readers will keep at hand for constant reference.

A. THOMSON

Quality in Flax

Presented by A. J. Turner. Pp. x + 249. Lisburn: Linen Industry Research Association. 1954. Price, 21s. 0d.

This is an account of work aimed at providing a complete and general solution of the problem of relating spinning quality to fibre properties, and thence specifying test standards from small lots of fibre. This necessitated investigating the effects of the various methods of retting and developing various methods of measuring the physical properties of fibres and their chemical composition. It is concluded that fibre (spinning) quality of flax can, in principle and with certain reservations, be estimated from measurements of fibre breaking stress, fineness, and length of fibre strands, but that it is impossible to set up as a standard any particular method of so doing. The book is not of general interest to tinctorial technologists, though those concerned with the processing of linen will be interested in Appendix II, which deals with the chemical composition of scutched fibre.

C. O. Clark

Technique of Organic Chemistry

Volume VI

Micro and Semimicro Methods

By N. D. Cheronis, A. R. Ronzio, and T. S. Ma. Pp. xxiii + 628. New York and London: Interscience Publishers. 1954. Price, $12.00.

The present volume is a worthy addition to this series of invaluable books (of which nine volumes have so far been announced), and it has been compiled by men of the widest possible experience and knowledge. Microchemistry has so developed that it is hard to credit that its origin, in the work of Emich during the first decade of this century, is so recent; even so, it remains largely a child of necessity. Few workers would resort to microchemical methods unless compelled by sheer shortage of material. The application of microchemical preparative methods by inexperienced workers has led to the establishment of an intermediate scale, which is now recognised in its own right as "semimicro"; it may be said to range from a few hundred milligrams to a few grams of material. Semimicro methods are of considerable value, especially in the educational field, in that they combine economy of materials with the requirement of a high level of practical discipline.

The introductory chapters include one in which the quantitative terminology of small-scale procedures is discussed; the confusing lack of general agreement is emphasised, and though excellent proposals are made for a reasonable and much-needed universal terminology, it is very doubtful whether this book (excellent as it is) will carry sufficient authority to be of much effect.

The book is divided into three parts—general (physical) methods, preparative reactions, and analytical procedures and reactions. The first part has four sections—crystallisation and collection of crystals; distillation, sublimation, and extraction; miscellaneous laboratory operations and tools; and measurement of physical constants. The vast experience of the author furnishes a useful guide through the maze of available apparatus and methods, and there is no lack of detail; the account of melting point determination using the Kofler hot-stage is remarkable in its thoroughness. Diagrams and photographs of apparatus are numerous, though tending to superfluity, e.g. the electric sand bath on p. 131.

Part II has the following sections—reduction; oxidation; halogenation; acylation, esterification, and hydrolysis; nitration and sulphonation; amination and diazotisation; use of organometallic compounds; dehydration, cyclisation, and condensations; and microsyntheses with tracer elements (which last includes an account of a contribution by a Russian (!) worker who is unknown to us—the "Breakoffsky" device). Here is a wealth—even an embarassment—of information presented in a way which bespeaks countless hours spent in bringing a welter of material from many scattered sources, to a very satisfactory state of organisation and presentation.

The sections of the last part of the book are—micromethods for the characterisation of organic compounds; tests for functional groups; preparation of derivatives (for which the term "derivatization" is proposed in view of its wide currency, though the equally ugly words "derivativization" and "derivativation" are considered to be of better parentage!); and quantitative micromethods for the estimation of functional groups. The most comprehensive and valuable section is that dealing with the preparation of derivatives, and it covers 93 pages and contains 280 references to the literature; the reviewer knows of no better review of the subject. As might be expected, the final section, on the quantitative microanalysis of functional groups, is in the nature of a brief summary of the current position; this is naturally due to the extensive treatment of the subject of functional group analysis in other works, notably the recent book by Siggia. Another subject to receive cursory treatment for the same reason is chromatographic analysis; together with adsorption, chromatography occupies the whole of Volume V in the *Technique of Organic Chemistry* series.

There are features in the compilation and editing which might be profitably revised in time for the second edition of this book (which must certainly come before long). The desire to treat quantitative terminology consistently, expressed on pp. 7–9, might be extended—thus, "inch(es)", "hour(s)", and "minute(s)" are written in full, though their abbreviations are the commonest of currency. On the other hand, "inside diameter" and "i.d.", and "outside diameter" and "o.d.", are used capriciously throughout the book. The prefix "micro" is attached with enthusiastic abandon to everything in sight, and hyphens would be a charitable gesture to the reader who is faced with such phenomena as "semimicrofractionating", "microebullioscopic", and "microvolumenometer". On the whole there are too many misprints for

comfort. The book is of such importance, and overall excellence, that attention to such apparent trivialities is well worthwhile.

The average Briton who vaguely imagines America as the home of Croesus, where scientists equip themselves with the best of everything—irrespective of cost—will be surprised to see that cost is taken into account, in detail, in many instances where a choice of method exists; many simple set-ups (the editor permits the luxury of a hyphen here) are described primarily because they can be contrived from inexpensive items that are available in most laboratories. Somehow the thought is comforting. J. W. DUARTE

British Standard

Recommended Names for Chemicals used in Industry

***B.S.* 2474 : 1954**

Pp. 63. London: British Standards Institution. Price, 10s. 0d.

This British Standard has been prepared by a Committee representing the Association of British Chemical Manufacturers, the Board of Trade, the Chemical Society, the Department of the Government Chemist, and the National Sulphuric Acid Association. All classes of chemicals used in industry have been considered, with the exception of dyes, pesticides, and the naphthol- and naphthylamine-sulphonic acids. The committee has been guided by the nomenclature used by the Chemical Society, but the recommendations do not entirely conform to this, account being taken of other systems and considerations.

The general rules adopted include the principle of "like treatment of like things" and the practically complete abandonment of the prefix "bi-". For organic chemicals, names of radicals which replace hydrogen are joined to the name of the parent compound, e.g. *nitrobenzene*; but generic names of classes of compounds are written separately, e.g. *diethyl ether*. *Oxo-* has been adopted as a prefix instead of "keto". For diethylene and triethylene glycols the trivial names *digol* and *trigol* have been coined. The name "cresyl" is to be abandoned, so that "tricresyl" phosphate becomes *tri*-m-*tolyl* phosphate. A single principle, viz. alphabetical order, is to be adopted in determining the order of naming substituents and metals in complex salts (with the exception of hydrogen in acid salts), e.g. *dodecyl sodium sulphate*; exceptions are made for the sake of clarity, e.g. *triphenylchloromethane*. The prefix "cyclo-" is not italicised, contrary to Chemical Society usage.

To denote the position of substituents, Greek letters and such prefixes as *ortho* are to be replaced by numerals, especially in the naphthalene series. However, exception is made for trivial names and for ring systems containing only two substituents. In spite of this, the recommendations include such unfortunate and confusing hybrids as "5-nitro-*o*-anisidine" and "4:6-dinitro-*o*-cresol".

The main body of this standard is occupied by a list of some 2,000 recommended, non-preferred, and [deprecated] names. Among those of special interest to readers of this *Journal* are—acetaldehyde sodium bisulphite, *p*-amino-*NN*-dimethylaniline [dimethyl-*p*-phenylenediamine], 4-amino-2′:3-dimethylazobenzene [*o*-aminoazotoluene], cellulose acetate and nitrate [acetyl- and nitrocellulose], chromic potassium sulphate [chrome alum], cuprammonium sulphate, cyclohexylamine, 1-diazo-2-naphthol-4-sulphonic acid, 1:2-dichloroethane [ethylene dichloride], 1:4-dihydroxyanthraquinone [quinizarin], glycerol [glycerine], iminazole, methylcellulose, 1-naphthol, phenazone [antipyrin], phenylazo-*p*-cresol [benzeneazo-*p*-cresol], potassium dichromate [potassium bichromate], sodium anthraquinone-2-sulphonate [silver salt], sodium carbonate [soda ash], sodium chloride [salt], sodium dithionite [sodium hydrosulphite], sodium 1-nitroso-2-naphthyloxide [sodium 1-nitroso-2-naphtholate], sodium pentachlorophenoxide [sodium pentachlorphenate], sodium sulphate [Glauber's salt], tetrachloroethylene [perchlorethylene], *o*-tolidine, and tolylene-2:4-diamine [toluylene-2:4-diamine].

Finally, recommended names are listed for grades of chemicals, e.g. seven each for sodium carbonate and sulphuric acid; the latter is at present ordered under nearly a hundred different descriptions! C. J. W. H.

Chemical Nomenclature

Edited by the staff of *Industrial and Engineering Chemistry*. Pp. v + 112. Washington, D.C.: American Chemical Society. 1953. Price, 22s. 6d.

This book, No. 8 in the Advances in Chemistry Series, is a collection of papers comprising the Symposium on Chemical Nomenclature, presented before the Division of Chemical Literature at the Diamond Jubilee meeting of the American Chemical Society held in New York in September 1951. Six countries are represented among the eleven papers, and the three nomenclature commissions of the International Union of Pure and Applied Chemistry are also represented.

In a letter of greeting the ninety-year-old A. F. Holleman of Amsterdam mentions that the I.U.P.A.C. in 1922 set up a commission to revise the Geneva nomenclature of 1892. Rules were framed for submission to the meeting of the Union at the Hague in 1928, but no decision was reached because individuals attempted to introduce their own schemes at the meeting. However, the proposals of the commission were approved at Liége in 1930.

A paper by W. C. Fernelius on the nomenclature of co-ordination compounds quotes several examples of the naming of metal-complex azo dyes, e.g. "*oo*′-azobis(phenoxido){*o*-(2-hydroxyphenylazo)phenoxido}cobalt (III)" for the 2 : 1 complex of *oo*′-dihydroxyazobenzene with cobalt.

K. A. Jensen of Copenhagen puts forward some unfamiliar ideas in a paper on *Problems of an International Chemical Nomenclature*. He pleads for the abandonment of national names (e.g. German "Wasserstoff" and "Bernsteinsäure", Danish "brint") and for a statement by the

I.U.P.A.C. that all new chemical names and terms should be derived from Greek or Latin roots (e.g. "zwitterion" to be replaced by *ampho ion*). Unification of the names of the chemical elements is to be achieved by—(*a*) introducing in scientific literature Latin names for the names of elements known from antiquity (e.g. *cuprum* for "copper"), and (*b*) abandoning duplicate forms (e.g. "sodium" in favour of *natrium*). Spelling should be etymological rather than phonetic (and often unrecognisable), so that the British "sulphur" should be *sulfur*, American "aluminum" *aluminium*, Scandinavian "krom" *chromium*, and Italian "esaossicicloesane" *hexahydrocyclohexane*. A uniform system of transliteration of words of Greek origin is required.

Papers by the editors of the *Journal of the Chemical Society* and of *Chemical Abstracts* stress the necessity for co-operation between British and American chemists in standardising chemical nomenclature. The importance of trivial names (a term used by Linnaeus) even in systematic organic chemical nomenclature is stressed by both F. Richter of the Beilstein-Institut, Hoechst, and P. E. Verkade of Delft. J. E. Courtois (of Paris), in *Work of Commission on Nomenclature of Biological Chemistry*, considers *inter alia* the nomenclature of carbohydrates, amino acids, and carotenoids. H. S. Nutting (Midland, Mich.), in *Nomenclature in Industry*, emphasises the economic value of a uniform and systematic nomenclature, especially in a large organisation. G. M. Dyson (of Loughborough) gives a brief historical survey of the *Development of Chemical Symbols and their Relation to Nomenclature*. Finally, J. W. Perry discusses in broad terms *The Rôle of Terminology in Indexing, Classifying, and Coding*, especially from the point of view of using punched cards and electronic devices in literature searches.

For those who are interested in the problems of chemical nomenclature, this collection of papers gives a valuable survey of the history of attempts at standardisation and of the difficulties which are still to be overcome. C. J. W. H.

Wool Research
1918-1954
Volume 3

Testing and Control in the Wool Industry

Pp. v + 277. Leeds: Wool Industries Research Association. 1955. Price, 25s. 0d.

This volume continues the publication of work done over many years at "Torridon". The majority of the subjects discussed are of only indirect concern to dyers and finishers, but the following chapter headings may be mentioned as being possibly of more direct interest—The Measurement and Control of Humidity in Testing Rooms; The Sampling of Wool Fibres (including dye sampling); The Measurement of Yarn Strength; The Continuous Strength Testing of Yarns; The Testing of Fabrics for Strength, Wear, Thickness, and Air Permeability; The Testing of Textiles for Water Repellency; Nomograms; Mill Experiments and Operational Research; The Determination of the Oil Content of Tops and Similar Material; and The Shrinkage Testing of Hosiery Fabrics and Garments.

A wealth of information is given in this volume, which contains 108 tables of data and 143 illustrations. C. J. W. H.

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University.

Binney & Smith & Ashby Ltd.

CARBON BLACKS FOR PROTECTIVE AND DECORATIVE COATINGS—This excellently produced loose-leaf book (pp. i + 45), written by L. J. Venuto, traces the history of carbon blacks from the 16th century B.C. to the present day, and points out that the Chinese makers of stick inks were the true discoverers of impingement carbon blacks, so important today.

Manufacture of carbon black began in earnest about 1860–1870 and now reaches 600 million lb. per year; if furnace blacks are included, the total production is in the region of 2000 million lb. per year, used mainly for the colouring of rubber but also for paints, lacquers, enamels, and printing inks.

The properties, physical forms, and uses of the various types are set out, and serve as a guide for the selection of the correct grade of carbon to use for any particular purpose.

Advice is given on the incorporation of carbon black into oils, resins, cellulose nitrate, varnishes, etc. as well as on the choice of solvents, diluents, and dispersing agents. The milling of carbon black is dealt with in detail, and recommendations for milling under different conditions are included.

Methods of testing blacks are outlined and suggestions made for overcoming possible difficulties.

This is a really informative publication, which will be of great value to present and potential users of carbon blacks. A. THOMSON

Ciba Ltd.

CATALYSER B CIBA—This is an organic compound recommended as a condensation accelerator—it provides the acid required for curing—in the application of thermosetting resins.

CIBANTINE OLIVE F2B—This solubilised vat dye gives dyeings of excellent fastness to light and wet treatments on all types of cellulosic materials. Somewhat brighter and bluer colours are obtained than with the Olive 2B brand, and the new product is particularly useful for producing green in conjunction with the various Cibantine Brilliant Greens. It is not sensitive to overoxidation. Fastness figures on cotton include—Light 8, washing (E.C.E.–*c*) 4–5, chlorine (E.C.E.–*b*) 4–5.

COPRANTINE NAVY BLUE BGLL and BRLL—These direct dyes give navy blues of good fastness to light and wet treatments when applied either by a single-bath method with Coprantine Salt II or by aftercoppering with either Coprantex B, Coprantine Salt II, or copper sulphate and acetic acid. The best fastness to washing is obtained with Coprantex B. Crease-resist finishing has no effect on the hue or the light fastness of the dyeings.

Dyeings may be discharged with Coprantine discharge paste. The fastness to washing of dyeings of the BRLL brand is slightly lower than is obtained with the BGLL brand. Both dyes are recommended for dyeing yarns, knitted fabrics, furnishings, and dress materials. Fastness figures for a dyeing on cotton produced by the Coprantine single-bath method include: *Coprantine Navy Blue BGLL*— Light 6–7, washing (E.C.E.–*b*) 4–5, perspiration 4–5. *Coprantine Navy Blue BRLL*— Light 6–7, washing (E.C.E.–*b*) 3–4, perspiration 4–5.

Dyeing Wool–Polyamide Fibre Yarns— This card contains 81 dyeings on 70 : 30 wool–polyamide fibre mixture yarns. These include dyeings on wool–nylon of 18 individual Neolan dyes and 18 mixtures, 18 level-dyeing acid dyes and 9 mixtures, and 9 Cloth Fast dyes. A further 9 dyeings on wool–Perlon of level-dyeing acid dyes are also included.

Dyes for Silk (Pattern Card No. 2036); Supplement to Pattern Card No. 2036— This is a particularly comprehensive card, containing (with the supplement) dyeings on silk cloth of 167 dyes classified as follows— level-dyeing acid 30, Cloth Fast 20, Neolan 27, Cibalan 24 (including 1 Cibalan Brilliant), direct 27, Chlorantine Fast 19, Diazo and Rosanthrene 16, and basic 4. A most useful feature is the series of tables intended to assist the dyer to select the most suitable dyes for his particular purpose. Fourteen different styles varying from evening gowns to flags and bunting are listed, and the minimum fastness specified by Section VIII of l'Association Internationale de la Soie (A.I.S.), Lyons, is specified for each. The suitability of a particular dye for dyeing on (*a*) unweighted and weighted and (*b*) unweighted silk only to meet these requirements may be determined by referring to the relevant table.

The Clayton Dyestuffs Co. Ltd.

Cibacet Dyes on Nylon— This card contains dyeings on filament nylon cloth of a range of 26 disperse dyes. There are one direct black and two blacks diazotised and developed with 2:3-hydroxynaphthoic acid. Diazotised and developed dyeings of three other dyes are also included, viz. Cibacet Oranges 2R and 4R and Cibacet Violet 5R. In the case of the orange dyes the colour changes to reddish violet, and the fastness to light is markedly reduced, but the fastness to hot pressing and wet treatments is much improved.

Imperial Chemical Industries Ltd.

Alcian Blue 8GX— This is a modified and improved brand of Alcian Blue 8G, the solubilised phthalocyanine derivative developed for printing cellulosic fibres. It may be applied by any of the techniques described in *Fast Printed Styles with Alcian Blue 8GS*. A non-volatile acid (lactic acid) and a hygroscopic solvent (Glydote B and/or glycerol) are added to the printing paste. During subsequent steaming the dye becomes insoluble, but only at a rate which also allows diffusion into the fabric. After steaming for 5–10 min. the bulk of the dye is fixed within the fibre. The remaining, water-soluble portion must also be fixed, but since it is present in the form of cations, this is readily achieved by treating the steamed material in a solution containing suitable dye-fixing anions, e.g. dilute acidified dichromates or aromatic sulphonic acids (Azoguard 35% Solution). Alcian Blue 8GX is compatible with other members of the Alcian X range. It is recommended only for printing cotton by the new technique; it is not dischargeable, and is not suitable for illuminated discharge styles. Fastness figures on cotton for a 2% print of Alcian Blue 8GX 300 include— Light 6–7, hypochlorite ("dry chemicking") 4–5, washing (Test No. 5, 5 times at 100°c.) 4.

Alcian Green 3BX and 2GX— These are temporarily solubilised printing dyes designed to give bright greens on cotton by direct printing and fixation by neutral steaming followed by a short treatment in an Alcian fixing bath (see above). They may be mixed in all proportions with other members of the Alcian X range and may be printed alongside vat, solubilised vat, Brentogen, basic, and mordant dyes. They may be printed on Brenthol-prepared grounds, and when fixed are fast to normal Brenthol-removal treatments, being better than most chrome greens in this respect. They are recommended only for printing cotton. They are not dischargeable, nor are they suitable for use as illuminating colours in discharge styles. Fastness figures for a 2% print on cotton include: *Alcian Green 3BX*— Light 6–7, hypochlorite ("dry chemicking") 5, washing (Test No. 5, 5 times at 100°c.) 4–5. *Alcian Green 2GX*— Light 5, hypochlorite ("dry chemicking") 4–5, Washing (Test No. 5, 5 times at 100°c.) 5.

Alcian Yellow GX— This temporarily solubilised printing dye is recommended for producing bright golden yellows on cotton. In printing characteristics and scope and limitation of application it is very similar to the other members of the Alcian X range, with which it may be mixed in all proportions to give a wide variety of colours by direct printing. The developing technique employed with this new range of dyes enables prints to be obtained having very good fastness to bleaching and repeated severe washing and much better fastness to wet and dry rubbing than was obtainable with the original Alcian Blue 8GS. Fastness figures for a 2% print on cotton include— Light 4, hypochlorite ("dry chemicking") 5, washing (Test No. 5, 5 times at 100°c.) 4–5.

Azoic Dyeing of Acetate Rayon— A reprint of a lecture by Dr. I. M. S. Walls (*J.S.D.C.*, **70**, 429 (1954)).

Caledon Yellow 3R— This vat dye gives reddish yellows of very high fastness to light on cellulosic fibres, and is particularly recommended alone and in mixtures for dyeing furnishing and casement fabrics, since it does not accelerate the tendering of the dyed material on exposure to light. It is best applied to cotton and viscose rayon by Method 2 at 50°c.; the leuco compound is moderately stable at 80–90°c., and dyeing at these temperatures for short periods does not cause serious dulling of the colour. All brands may be applied by leuco-padding; FD Powder Fine and Paste Fine brands are recommended for pigment padding and the Abbot–Cox process. Fastness figures on cotton include— Light 7, soda boiling (open kier) 4, hypochlorite 4–5, washing (Test No. 5, 5 times at 100°c.) 4–5.

Fixanol PN in the Textile Industry: Auxiliary Products Pamphlet No. 91— This comprehensive pamphlet includes the following sections— general properties of Fixanol PN; application to cellulosic fibres, including continuous processes; the fastness characteristics of dyeings on cotton and viscose rayon of direct dyes aftertreated with Fixanol PN; use in dyeing rayon-staple cloth subsequently to be crease-resisted; use on wool–cellulosic fibre mixtures, on nylon–cotton hosiery, in dyeing bast fibres, and in textile printing.

Lissapol N for the Scouring of Worsteds and Woollens: Auxiliary Products Pamphlet No. 75.

Problems in the Dyeing of the Newer Synthetic Fibres— A reprint of a lecture by Dr. I. M. S. Walls (*J. Textile Inst.*, **45**, P 258 (1954)).

Scutamol and the Control of Slime in Paper Mills— This Technical Circular contains an introduction to the subject of slime growth in paper-mill water systems and describes the use of an organic mercurial preparation, Scutamol, in controlling it.

Technical Information Leaflets— *Dychouse No. 175. The Dyeing of Orlon*— Information is given for dyeing Orlon Type 42 staple fibre using disperse and basic dyes and acid dyes by the cuprous-ion technique. In each case a list of suitable dyes is given together with the daylight fastness of dyeings of medium depth.

Dychouse No. 176. The Dyeing of Acrilan— Acid, chrome, metal-complex (Ultralan), disperse, and solubilised vat dyes are recommended. The last-named give dyeings having the best wet fastness and in many cases high fastness to light.

Dychouse No. 182. The Dyeing of "Dynel"— Selected disperse dyes, acid dyes applied by the cuprous-ion technique, basic dyes, and solubilised vat dyes are recommended; the last-named are particularly suitable for pale dyeings of excellent wet fastness and good fastness to light. Attention is drawn to those characteristics of dynel which are of importance to the dyer— (1) it softens in hot water; (2) it is delustred at or near 100°c. but may be relustred by treatment in boiling sodium sulphate solution or by dry heat; (3) it shrinks appreciably at or

near 100°C., and shrinkage is promoted by "carriers"; (4) it turns brown on prolonged exposure to sunlight.

Dyehouse No. 231. The Dyeing of "Terylene" Polyester Fibre–Wool Unions— This details slight modifications of the original process, found to be desirable in the light of bulk experience. A selection of suitable dyes is given for the Terylene component, with details of their partition properties and the fastness of the dyeings to light and sublimation.

Dyehouse No. 232. Basic Dyes for Dyeing Orlon and Orlon Unions— This gives further details of the dyeing behaviour of basic dyes. Ten dyes are specified, and the amount of each absorbed at 100°C. in 1 hr. is given together with the daylight fastness for a medium-depth dyeing. Recommendations are made for dyeing Orlon–wool, and Orlon–nylon, and Orlon–cellulosic fibre mixtures. For the former basic dyes are used to dye the Orlon; using selected dyes at 100°C. with 10% of acetic acid (30%) and dyeing for 1–2 hr., it is possible to produce dyeings of light to medium depth on the Orlon with little or no staining of the wool. Solid dyeings or multicolour effects may be obtained by dyeing the wool first with acid milling dyes. Basic dyes dyed with 1% acetic acid at 100°C. are also recommended for the Orlon in Orlon–nylon mixtures. In this case the nylon can subsequently be dyed with disperse dyes at 80°C. Solid blacks are also stated to be possible using the cuprous-ion technique and bringing the less heavily dyed nylon up to shade with disperse dyes. With Orlon–cellulosic fibre blends, disperse or basic dyes may be used on the Orlon, the cellulosic portion then being dyed with direct or solubilised vat dyes.

Dyehouse No. 235. Disperse Dyes for Dyeing "Orlon" and "Orlon" Unions— This note is complementary to Dyehouse No. 232. The value of disperse dyes for producing pale dyeings of good fastness to light is illustrated. The fastness to washing and rubbing is also good, and in the case of medium-depth dyeings is improved by an aftertreatment in Lissapol NC (2 g./litre) for 15 min. at 50–60°C.

Dyehouse No. 237. The Dyeing of "Terylene" Polyester Fibre: Supplement No. 2. A New Process for Blacks on Loose Staple, Slubbing, and Yarn— An azoic black based on Dispersol Diazo Black B and Brenthol OT has been found to produce good blacks having high fastness to rubbing and light. There is an appreciable saving in time and in the quantities of dyes and auxiliaries required. A dyeing temperature of 120–130°C. is essential. The Dispersol Diazo Black B is applied first at 120°C. for 10 min., the dispersed Brenthol OT then added, and dyeing continued at 125–130°C. for 30 min. Development is followed by a "reduction-clear" and soaping in the usual manner.

Dyehouse No. 239. The Dyeing of "Terylene" Polyester Fibre: Selection of Disperse Dyes (Supplement No. 3)— Two selections of dyes are given. The first group of 5 dyes is particularly recommended for dyeing 100% Terylene staple, slubbing, and yarn where good fastness is required to sublimation and pleating by either the dry-heat or the steaming process. Dyeing at 100°C. using Tumescal D or *o*-phenylphenol, or at 130°C., is recommended. Recipes are provided for matching 11 B.C.C. shades; the dyeings are of fastness 5–6 or better to light. Where the dyed material will not be subjected to high-temperature treatments and maximum fastness to light and wet treatments is not essential, a second group of 6 dyes is recommended which dye Terylene more readily.

Dyehouse No. 243. The Dyeing of Cellulose Triacetate Fibres— Cellulose triacetate is not damaged by ironing at 220°C. or by dyeing at 130°C. at pH < 10. Disperse, azoic, and selected vat dyes appear to be the most useful; basic, water-soluble acetate-rayon, and solubilised vat dyes are of no interest at present. Dyeings of disperse dyes are faster to wet treatments than the corresponding dyeings on acetate rayon. Dyes sensitive to "gas-fume fading" on acetate rayon are even more sensitive on the triacetate. This class of dye is completely stripped in cold trichloroethylene. Azoic dyeings may be produced by the "modified" process; the combination Brenthol OT–Dispersol Diazo Black B is valuable for fast blacks. Selected indigoid vat dyes may be applied from solutions of their leuco compounds buffered at pH < 10; pale to medium dyeings only are suggested at present; these have excellent fastness to light and wet treatments.

Dyehouse No. 245. Weathering Fastness: Vat and Azoic Dyes— Dyeings of many vat and azoic dyes show much lower light fastness when exposed under the alternate wet and dry conditions of normal outside exposure (weathering). A large number of standard-depth dyeings of vat dyes and deep dyeings of azoic combinations have been exposed to radistion from a carbon arc while being sprayed intermittently with water (cf. *Hexagon Digest* No. 9): B.S.I. light-fastness standards for comparison were exposed simultaneously but under dry conditions in a Perspex box. Notable results among the vat dyes include— Caledon Yellow 5G 1, Caledon Gold Orange 3G 3, Caledon Jade Green 4G 3–4.

Rubber No. 17. The Scorching Characteristics of Kosmos 45 Black— Vulcafor HBS and Vulcatard A as a Safe Curing System.

Rubber No. 18. Vulcaprene Synthetic Polymer.

Rubber No. 19. Flock-sudding with Vulcaprene Adhesives.

Rubber No. 21. Varnishes based on Vulcaprene for Cured Rubber Articles.

E. I. du Pont de Nemours & Co.

SEVRON YELLOW L— This is a basic dye (formerly known as "Du Pont Basic Yellow OL") particularly designed for dyeing Orlon type 42 acrylic fibre. It is marketed in the U.K. by Brown & Forth Ltd. It gives brilliant yellows of high fastness to light and wet treatments, and may be dyed either at 100°C. or at temperatures up to 110°C., when better exhaustion is obtained. It is also of interest for dyeing wool–Orlon mixtures; under faintly acid conditions the staining of the wool may be reduced to very small proportions; and multicolour effects can be produced by a single-bath technique. Sevron Yellow L is also of interest for dyeing silk and nylon and for both dyeing and printing acetate rayon. Fastness figures on Orlon type 42 include— Light (Fade-Ometer exposure) 6–7, washing (AATCC 3) 5, milling (4½ hr. at 32°C.) 5.

SEVRON RED 4G— This is a basic dye (formerly known as "Du Pont Basic Red 4G") belonging to the special range introduced for dyeing Orlon type 42 acrylic fibre. It gives brilliant, fluorescent scarlets having excellent fastness to wet treatments, and is recommended for use alone and in mixtures with other members of the Sevron range. It may be dyed at the boil or above 100°C. under pressure. Since wool may be left unstained and cellulosic rayons only slightly stained, it is of particular interest on wool–Orlon and cellulosic fibre–Orlon mixtures, multicolour effects being readily obtainable. It is also suitable for dyeing silk and nylon and for dyeing and printing acetate rayon. Fastness figures on Orlon type 42 include— Light (Fade-Ometer exposure) 4–5 (N.B. This figure actually represents the fastness of the rather more bluish colour to which the dyeing reverts after a comparatively short exposure (5 hr.) but which thereafter alters only slightly during a much longer period), washing (AATCC 3) 3, milling 5.

SEVRON BLUE B— This is yet another basic dye (formerly "Du Pont Basic Blue OB") specially designed for producing light to medium blues on Orlon type 42. It may be applied at 100°C. in the presence of acetic acid and sodium acetate or at temp. > 100°C., when brighter dyeings and better exhaustion are obtained. Like other members of the range, under suitable conditions the staining of wool and cellulosic fibres is reduced to a minimum, and thus multicolour effects may be produced by single-bath dyeing techniques. Efficient scouring after dyeing improves the brightness and wet fastness of dyeings both on Orlon and on Orlon–wool and Orlon–cellulosic fibre mixtures. It has little affinity for acetate rayon and nylon but is of interest for medium blues on silk. Fastness figures on Orlon type 42 include— Light (Fade-Ometer exposure) 6–7, washing (AATCC 3) 5, milling 5.

Sandoz Products Ltd.

ALIZARIN LIGHT BLUE HRL— This equalising acid dye is very similar in dyeing properties and fastness characteristics on wool to Alizarin Light Blue 4GL but is much redder in hue. It is recommended alone and in mixtures for dyeing yarn and cloth and particularly wool and fur felts. On polyamide fibres it will build up to medium depth. It may be applied at high temperatures (108°C.) without change in hue or loss in depth. Cotton, viscose rayon, and acetate rayon are unstained. Fastness figures on wool include— Light 6, washing (40°C.) 3–4, perspiration 2–3.

SANDOZOL OLIVE-GREEN 2B— This solubilised vat dye is recommended for dyeing and printing all types of cellulosic fibres. The colours obtained are bluer than those given by the Olive-Green B brand, and not sensitive to overoxidation. The very high fastness of dyeings and prints to light and wet treatments makes it very suitable for furnishings and coloured woven goods. It may be applied by either the one- or the two-bath method on the jigger or winch or in circulating-liquor machines, or by padding, and can be developed hot or cold. Fastness figures on cotton include— Light 8, washing (95°C.) 4–5, chlorine 4–5.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

Design Features of a Pilot-plant Continuous Range for Chemical Treatment of Cotton Yarn and Fabric. E. J. Keating, A. S. Cooper, A. L. Murphy, and H. O. Smith. *Amer. Dyestuff Rep.*, **44**, 65–68 (31 Jan. 1955).

A group of basic finishing units is capable of being assembled in a number of ways and is thus able to handle various experimental finishing treatments. It comprises 5 combination padder–washer units each consisting of a 3-roll padder in the centre and a washer section at each end, a hot-air dryer, and a batcher. By adding a cooling cylinder, feed tanks, and an exhaust hood, the machine can be used for the partial acetylation of cotton.

J. W. B.

Treating Cotton with Volatile and Toxic Chemicals. L. H. Greathouse, C. H. Haydel, and H. J. Janssen. *Ind. Eng. Chem.*, **47**, 187–191 (Feb. 1955).

A sealed pilot-plant reactor unit is described which is suitable for treating fibre, yarn, or fabric under controlled conditions of pressure, temperature, and reagent flow. The unit has been used successfully to cyanoethylate 50 yd. of cotton cloth and to decrystallise with ethylamine 5 lb. of lint. To aid the selection of suitable constructional materials, the resistance of various metals, tubings, gasket materials and sealing compounds to ethylamine, acrylonitrile and acetic acid–anhydride acetylating reagent, was evaluated and the results are tabulated.

W. K. R.

PATENTS

Thread-advancing Thread-storage Device. Courtaulds. *BP* 723,779–80

In a thread-advancing thread-storage device on which thread is treated with several liquids means are provided to prevent the liquids flowing into each other. C. O. C.

Treating Filaments, Threads, Tapes, etc. P. L. Pendleton and B. R. Sturges. *USP* 2,665,699

Modification of *USP* 2,612,860.

The thread passes through a channel forming part of a vibrating member without touching the sides of the channel. This member is mounted in a liquid with the liquid level partly above and partly below the channel area. The vibrations generate in the channel a fluid wave which rises above the normal liquor level and which is dispersed in the channel as an elevated field of reciprocating fluid particles. These particles bombard the strand passing through the channel. C. O. C.

Reducing Transverse Movements of Filaments during their Continuous After-treatment. Vereinigte Glanzstoff–Fabriken. *BP* 725,955

When continuously wet-processing filaments, e.g. after spinning, transverse movement of the filaments is greatly reduced by having on both sides of the group of filaments walls extending at least up to the surface of the bath. These walls must extend close to the guide rollers and be close to the inter-travelling filaments. C. O. C.

Continuous Wet Treatment of a Number of Threads. Algemene Kunstzijde Unie. *BP* 725,970

A number of parallel, separated threads pass through a trough having a guiding roller at its beginning and an inlet and outlet for the liquid at its end. The inlet and outlet are so arranged that the liquid in the trough is some millimetres, e.g. 3–7 mm., above the bottom of the guide rollers. There is a plate placed 1–5 mm. below the guide rollers. The threads pass over this plate and carry some of the liquid with them in a path only slightly wider than that of the threads thus preventing back and cross currents in the liquor in contact with the threads.

C. O. C.

Drying Rayon Cakes or Similar Packages. American Viscose Corpn. *BP* 725,668

Irregularities and differences in residual shrinkage, dyeing and other properties can be greatly reduced by correlating the rate of dielectric heating to the conditions prevailing around the package so that the rate of vapour emission is at least sufficient to keep a layer of the atmosphere immediately surrounding the package saturated with the vapour evolved at the temperature of drying. This can be done either by heating the package so rapidly that the vapour is developed so quickly that it replaces the atmosphere surrounding the package as fast as it is cleared away by normal convection currents, or by placing a cover about the package so that a layer of atmosphere saturated with the vapour at the temperature of drying is retained around the package. C. O. C.

Top and Yarn Drying. S. A. Orsi Mangelli-Raion Forli. *BP* 724,701

Where material is being dried by following a helical path around a cylinder which is heated by hot air being circulated in a helical path between the drying surface and an inner shell, steam is used to heat the air in a closed cycle so that it passes from a heat exchanger to the cylinder and returns again to the heat exchanger. C. O. C.

Drying Tunnels for Textiles. Courtaulds. *BP* 724,057

A drying tunnel, e.g. for rayon cakes, is made up of several contiguous regions each of which has an external duct housing a fan and a heater and connected across opposite sides of the region to circulate a current of air transversely across the region. An inlet controlled by a damper opens into the duct at the fan to admit fresh air as desired into the air current. Stale air escapes from an outlet in the duct at the same rate as fresh air is admitted. A vent to the atmosphere communicating with the outlet maintains the pressure of air in the outlet at atmospheric pressure. Thus the humidity in each region can be independently controlled and there is no movement of air from region to region in the tunnel. C. O. C.

Centrifuges for Spools of Yarn. H. Frauchiger. *BP* 725,351

Wet Processing of Granular or Fibrous Material. Aktiebolaget Pluria. *BP* 724,027

The material, e.g. yarn, is placed in a circulation system into which the treating liquor is fed. A vacuum is created within the system and is then lifted to cause the liquor to be sucked into and among the material to be treated. The amount of liquor fed into the system is just enough to cover the material after the vacuum has been lifted. After the vacuum has been lifted all liquor in excess of

that retained by the material and required for treating it is removed. Finally a stream of air is circulated through the system so as to nebulise the liquor retained by the material and partly dry it. The air laden with the particles of liquor is passed through a device to remove them and is then re-used. C. O. C.

Nylon Hosiery Pre-steaming Cabinet. Paramount Textile Machinery Co. *BP* 723,612

Nylon hosiery is treated in a cabinet preheated by radiant heating coils and warm air circulated through the material to avoid condensation during subsequent injection of steam. Steaming at atmospheric pressure is followed by passage of hot air to dry the material. The various cycles of operation are automatically timed and the machine enables accurate control of conditions within the chamber. G. E. K.

Compensating Rolls for Handling Roped Fabrics, etc. Proctor & Schwartz. *BP* 724,462

Equipment in which the material is continuously treated with automatic allowance for changes in the rate of progress of the fabric, this being effected by use of one or more handling rolls which are tapered to provide varying rates of handling of the material according to its point of engagement along the length of the roll. C. O. C.

Coating Wire with Viscous Liquid. Connollys' (Blackley). *BP* 724,952

Apparatus for Coating Wires, Threads, etc. British Insulated Callender's Cables. *BP* 724,760

Fabric Shrinking. Famatex. *BP* 722,935

The fabric is alternately moistened and dried several times in an unbroken operation while it is held by the selvedges in such a manner that the tension in either warp or weft can be predetermined. C. O. C.

Roll-handling Device. M. E. P. Magaud. *BP* 723,763

The roll is mechanically loaded on to a truck and is then directly loaded on to the support arms of the machine in which it is to be used. C. O. C.

Fabric etc. Drying. Aktiebolaget Svenska Fläktfabriken. *BP* 725,048

Modification of *BP* 668,741 (J.S.D.C., **68**, 216 (June 1952)). Several blast boxes with discharge orifices alternating with outlet passages for the drying medium are arranged vertically over each other and placed in groups side by side in a closed chamber. The fabric passes alternately over and under rollers placed at alternate series of blast boxes. This arrangement obviates deflection of the fabric, makes it possible to place the blast boxes very close together and as the drying or conditioning medium is discharged closer to the fabric it is possible to increase the heat transmission between the drying medium and the fabric. C. O. C.

Fabric Drier. J. O. Ross Engineering Corpn. *USP* 2,671,279

A drier for crêpe fabrics in which ducts under the conveyor project air against the fabric and lift it off the conveyor. Other ducts placed above the fabric and between the lower ducts deliver air which forces the fabric back on to the conveyor. Thus each side of the fabric has drying air flowing against it throughout the greater portion of its passage through the drier. C. O. C.

Combined Clip and Pin Strip for Tenters. Lindauer Dornier Ges. *BP* 725,229

In a combined clip and pin strip when the pin strip is not in use it is put under a clamping plate having a carrier for the pin strip. When the pin strip is in use it is placed in front of the clamping plate. C. O. C.

Spraying Atomised Liquid on to a Moving Web. Vapo Systems Co. *USP* 2,672,844

The liquid is sprayed on to the web from a number of atomising nozzles each fed from two parallel pipes one of which supplies compressed air and the other the liquid. C. O. C.

Adjustable Doctor Blade for Coating Machines. Western Electric Co. *USP* 2,672,119

Apparatus for Applying Metal Powder to Sheets or Webs. W. Müller. *BP* 725,884

A scoop- or pick-up roller forms the base of the container for the metal powder. A number of plates moveable independently of each other towards and away from the roller define an outlet of variable size between the edges of the plates and the surface of the roller. C. O. C.

Fabric Printing Machine. A. J. C. de O. Barros. *BP* 725,885

The printing cylinders each have a partly permeable printing surface and an internal feed of colour. They are applied to an endless belt whose length is equal to either the sum or a multiple of the combined diameters of the cylinders. The belt is held against each cylinder by freely rotating rollers. The fabric is fed on to the belt and is presented to each cylinder along the common line of tangency of the cylinder, the belt and the roller carrying the latter. This contact is effected in a zone or curvature considerably more accentuated than with a pressure roller, and consequently in a narrower zone, resulting in a better and clearer impression than would be obtained by use of a pressure roller. As the same parts of the belt always come in contact with the same part of the printing cylinders there is no danger of soiling and so a liner is unnecessary. C. O. C.

Garment Washing and Drying Machine. C. B. Dodge. *BP* 723,135

Efficient use of the heat consumed combined with good handle of the dried goods is obtained by subjecting the interior of the chamber alternately to a current of hot air and to reduced and atmospheric pressure. C. O. C.

Laundry Washing and Similar Machines. Isaac Braithwaite & Son Engineers. *BP* 723,660

In a drum washing machine provided with an external liquid circulating system in series with the machine, an external tank in the circulating system has a float which controls a valve in the fresh liquid supply pipe to the machine so that the supply of liquid is stopped when it reaches a predetermined level in the machine. Manually operated means prevent circulating of the liquid through the tank and machine and allow fresh liquid to travel through the machine and then pass to waste. C. O. C.

Working Atmospheres containing Hydrogen Sulphide and Carbon Disulphide (particularly in Viscose Spinning) (VI p. 264).
Coating Cloth and Paper with High Melting Point Thermoplastic Material (X p. 271).

II— WATER AND EFFLUENTS

Treatment of Water for Textile Uses. O. Reichling. *Melliand Textilber.*, **36**, 79–83 (Jan. 1955).

A review of the filtration, coagulation, and softening processes available. S. R. C.

PATENTS

Boiler Composition. B. F. Goodrich Co. *BP* 724,683

A mixture of 0·5–50% by wt. of a polyacrylate and 99·5–50% of an alkali metal carbonate and/or bicarbonate and/or orthophosphate and/or polyphosphate is used so as to provide 0·25–5·0 p.p.m. of polyacrylate in the boiler feed water. It maintains the sludge in a fluid or gelatinous form having no tendency to crystallise. C. O. C.

Removing Boiler Scale, etc., by Use of Ion-exchange Materials. Phillips Petroleum Co. *USP* 2,671,035

Boiler scale and scale in tanks, pipes etc., may be removed by passing an aqueous slurry of an ion exchange material, e.g. a zeolite, through the boiler or pipe and in contact with the scale. C. O. C.

III— CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

French Synthetic Detergents from Petroleum. G. C. Inskeep and A. Mussard. *Ind. Eng. Chem.*, **47**, 2–10 (Jan. 1955).

A description is given of a new French plant for the large-scale production of the *sec.*-alkyl sulphate, Teepol (Shell), by the direct sulphation of olefins derived from cracked paraffin waxes. W. K. R.

Bactericidal and Bacteriostatic Powers of Detergents. J. Vallée. *Teintex*, **20**, 92–101 (Feb. 1955).

At 20°c. anionic and non-ionic detergents do not exceed soap in bactericidal action, compared at 1 g. of active ingredient per litre and 15 min. wash. Under the same conditions at 60°c. a positive bactericidal action on *Staphylococcus aureus* and *Bacillus coli* is evident. At 90°c. a positive action against *B. subtilis* develops, but the temp. alone is sufficient to kill the other two organisms. No connection between the bactericidal power and the pH of the synthetic detergents was noted. The latter were normally found to be sterile in the concentrations as sold, as was 72% cake soap. S. R. C.

Organic Additives and Lowering of Surface Tension. E. J. Burcik and R. C. Newman. *J. Colloid Sci.*, **9**, 498–503 (Dec. 1954).

Addition of 1-dodecanol or dodecanoic acid to solutions of sodium dodecyl sulphate lowers surface tension and causes a decrease in the rate of surface tension lowering. Addition of *n*-decane does not produce these effects. It is suggested that dodecanol and dodecanoic acid associate with the sodium dodecyl sulphate. W. R. M.

Ageing and Retrogradation of Starch Solutions— I. M. Ulmann. *Kolloid-Z.*, **140**, 32–41 (Jan. 1955).

Chromatographic analysis of starch soln. by means of an Al_2O_3 column enables changes in the systems amylopectin–H_2O and amylose–H_2O to be detected alongside each other. These changes are found to occur during the ageing of dil. starch soln. and depend upon experimental conditions, data for which are now recorded. Various colloidal phenomena are described, and conditions are obtained for precipitations of amylose contaminated with traces of amylopectin. The degradation processes occurring during ageing produce dextrins together with cleavage products which give no colour with iodine. H. H. H.

Mothproofing Agents. XIV— Relationship between the Chemical Constitution of Mothproofing Agents and their Effects on Wool. K. Matsui. *J. Soc. Org. Synthet. Chem. Japan*, **11**, 393–399 (1953): *Chem. Abs.*, **49**, 971 (25 Jan. 1955).

A large number of compounds of formula [3:5:2-Cl_2(OH)-C_6H_2]$_2$CHR have been synthesised and applied to wool which was then exposed to *Attagenus piceus*. When R = C_6H_5, Cl, NO_2, NH_2, or $CH_2NC_5H_4$, the mothproofing effect is low. Introduction of a sulphonic acid group into the benzene ring increases the mothproof effect considerably: it appears to impart greater affinity for the fibre, and the increased water-solubility permits ingestion of the compound by the larvae. C. O. C.

PATENTS

Improving the Foaming Properties of Detergents. Lever Brothers & Unilever. *BP* 711,467

Compounds of formula R^1·Ar·O·R^2·OH (R^1 = straight or branched chain hydrocarbon containing 4–10 C and at least one straight chain of > 4C, or a cyclic satd. hydrocarbon of 5–10C; Ar = benzene or naphthalene; R^2 = alkylene, a dialkylene ether or an alkylene polyether of < 16C and < 40 nor > 5C in any one uninterrupted C chain), e.g. *p-tert*-octylphenylethylene glycol ether, greatly improve the foaming properties of organic ionic soapless detergents. C. O. C.

Alkoxyalkylamine Salts of Alkylaryl Sulphonic Acids— Wetting Agents and Detergents. Atlantic Refining Co. *USP* 2,673,215

The γ-alkoxypropylamine salts of alkylarylsulphonic acids while less soluble than many known liquid detergents are markedly better detergents for proteinaceous and fatty soils. C. O. C.

Surface Active Agents. Monsanto. *USP* 2,673,842

Mixtures of alkali metal or ammonium sulphates of 2-butyloctanol-1-polyglycol ethers and alkali metal or ammonium salts of *N*-alkyltaurines (Alk of 12C) have markedly superior lathering properties to either constituent when used alone. C. O. C.

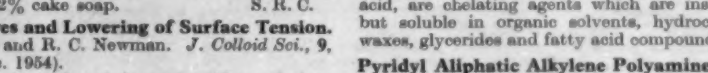
Substituted Alkylene Polyamine Acetic Acid Compounds— Chelating Agents. Dow Chemical Co. *BP* 723,316

Compounds of formula MOOC·CRH·N(CH$_2$(COOM))·A·(N(CH$_2$·COOM)·A)$_n$N(CH$_2$COOM)CHRCOOM (R = subst. or unsubst. Alk of 1–18C; A = CH_2·CH_2, CH_2·CH·CH_3, C_2H_5·CH·CH_2 or CH_2·CH_2·CH_2; n = 0, 1 or 2; M = H, NH_4, K, Na or an amine), e.g. ethylene diamine-bis-alanine-*NN'*-diacetic acid, are soluble in polar and non-polar organic solvents and in mixed aqueous and organic systems. They have strong chelating properties. *BP* 723,317

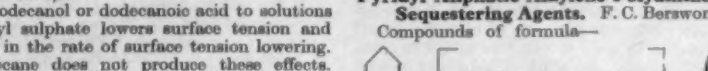
Compounds of formula CH_2RN·A·(N(CH_2COOM)·A)$_n$·NRCH_2COOM (R = Alk of 7–18C when A = polylene, butylene or trimethylene or R = Alk of 9–15C when A = ethylene) M = H, NH_4, K, Na or an amine; n = 0, 1 or 2), e.g. *NN'*-di-dodecyl ethylene diamine diacetic acid, are chelating agents which are insoluble in water but soluble in organic solvents, hydrocarbon oils and waxes, glycerides and fatty acid compounds. C. O. C.

Pyridyl Aliphatic Alkylene Polyamine Poly Acids— Sequestering Agents. F. C. Bersworth. *BP* 726,268

Compounds of formula—



(R = sat. chain of < 13C or low mol. wt. Alk; Alkylene = ethylene, propylene or trimethylene; n = 1 or 2; x = 1, 2 or 3), e.g.—



have great affinity for alkaline earth and rare earth metals and for a large number of other metal ions which are difficult to chelate, e.g. Pb, V, Zr and U. Those where n = 1 have better chelating properties than those where n = 2. C. O. C.

Sequestering Agents. F. C. Bersworth. *USP* 2,673,213

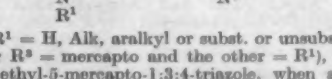
Compounds of formula (HOZ)$_2$N·X·NA·Y·NA·X·N(ZOH)$_2$ (Z = straight or branched chain bivalent alkylene of 2–6C; X = bivalent alkylene of 4–12C which may be interrupted by ether O atoms; A = CH_2COOH, CH_2CH_2·COOH, CH_2·PO(OH)$_2$, CH_2CH_2PO(OH)$_2$, $CH_2CH_2SO_3H$ or the alkali metal or ammonium salt of one of these groups; Y = bivalent alkylene of 2–3C), e.g. *NN'*-di-(2-iminodiethanolethoxyethyl)-ethylene diamine diacetic acid, are metal ion sequestering compounds whose end amino N atoms form acid salts with mineral and fatty acids.

USP 2,673,214

Compounds of formula (HOCH$_2$CH$_3$)N·CH_2·CHOH·(CH_2)$_n$·NA·Y·NA·(CH_2)$_n$·CHOH·CH_2·N(CH_2CH_2OH)$_2$ (n = 1–4; A = CH_2COOH, CH_2CH_2COOH, CH_2PO_3H, CH_2·CH_2PO_3H, $CH_2CH_2SO_3H$ or an alkali metal salt of one of these groups; Y = bivalent alkylene of 2–3C and may bear one or more Alk of 1–2 C), e.g. *NN'*-di-(2-hydroxy-3-iminodiethanol propyl)-ethylenediamine-*NN'*-diacetic acid are metal, particularly ferric ion, sequestering agents active over a wide pH range. C. O. C.

Inhibiting the Tarnishing of Metals by Detergents. Ilford. *BP* 722,331

Triazoles of formula—



(R^1 = H, Alk, aralkyl or subst. or unsubst. Ar; either R^2 or R^3 = mercapto and the other = R^1), e.g. 1-phenyl-2-methyl-5-mercapto-1:3:4-triazole, when added to detergents, prevent the tarnishing of brass, copper and similar non-ferrous metals or their alloys. C. O. C.

Textile Lubricants. Celanese Corpn. of America. *BP* 725,799

A stable, self-emulsifying lubricant, e.g. for oiling a tow of acetate rayon to be converted into staple fibre, comprises (A) a partial ester of phosphoric acid with a long chain fatty alcohol, (B) an alkylolamine–long chain fatty acid condensate, (C) an alkylolamine or tertiary amine, (D) a polyoxyethylene ether of a long chain fatty alcohol or a polyoxyethylene ester of a long chain fatty acid, (E) an alkyl phenol, all blended into (F) mineral oil. C. O. C.

Permanent Waving Solution. Eau de Cologne fabriek J. C. Boldoot. *BP* 722,328

An aqueous solution for use at room or body temperature has pH 6–7 and contains an alkali metal and/or ammonium sulphite and/or bisulphite and water-soluble ethers and/or esters which initially inhibit swelling of the hair, e.g. ethylene glycol monomethyl ether acetate and diethylene glycol monoethyl ether. The solution may also contain a swelling agent, e.g. ammonium thiocyanate. C. O. C.

Permanent Waving Composition. Gillette Co. *BP* 723,917

Thiols, e.g. ammonium thioglycollate, sodium mercaptoethanesulphonate or 3-mercapto-2-hydroxypropane sulphonic acid, when used at pH 8·6–9·6 not only reduce the disulphide bonds but also cause substantial swelling of the hair. This swelling is as important in permanent waving as is the reduction of the disulphide bonds, thus cysteine does not normally give an acceptable wave but in presence of urea it becomes as effective as ammonium thioglycollate. Addition of urea and certain of its derivatives, e.g. methyl or ethyl urea but not polyalkyl or aryl ureas, enhances the effect of all thiols. C. O. C.

Aqueous Dispersions of *N*-Alkoxymethyl Polyamides as Textile Finishes. DuP. *BP* 722,774 *BP* 723,952

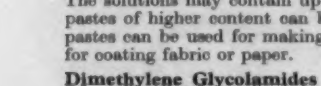
Aqueous dispersions of *N*-alkoxymethyl hexamethylene adipamides made with the aid of salts of a nitrogenous base and an acid of ionisation constant for the first or only H atom of $< 1 \times 10^{-3}$ at 25°C. and which decompose at < 170°C., e.g. NH_4 citrate, are useful stable textile finishes which are self curing on being heated. C. O. C.

Solutions of Elastomers derived from Diisocyanate-modified Polyesters—Filaments, Coating Compositions, etc. FBy. *BP* 722,723

The products obtained according to the processes of *BP* 700,608 and 700,611 by treating linear polyesters with diisocyanates and then cross-linking by treatment with water, dihydroxy compounds or diamines yield stable solutions in the solvents used for polyacrylonitrile especially in dimethyl formamide, dimethyl acetamide, dimethyl propionamide and methoxydimethyl acetamide. The solutions may contain up to 50% of the polymer or pastes of higher content can be made. Such solutions or pastes can be used for making filaments or films or used for coating fabric or paper. C. O. C.

Dimethylene Glycolamides as Solvents for Acrylonitrile Polymers. DuP. *USP* 2,671,067

Compounds of formula—



(y = a whole number; x = such that x/y is > 1 except that when the amide N atom is part of a saturated ring x/y is > 2; all the valencies except the carbon to carbon linkages and not shown as satisfied are satisfied by H, Hal, O, bivalent S, OH, thiol, cyano, thiocyano and sulphoxy, the total number of Hal, O, S, OH and thiol being $> y$ and of cyano, thiocyano and sulphoxy $> 2y$) e.g. *NN*-dimethylglycolamide, are good solvents for polymers containing < 85% of acrylonitrile. C. O. C.

Mixtures of Nitric Acid and Nitromethane as Solvents for Polyacrylonitrile. P. Halbig. *USP* 2,671,768

Mixtures of nitric acid and nitromethane dissolve polyacrylonitrile at room temperature to yield colourless solutions stable for several days and from which the polyacrylonitrile can be recovered without any change even after a long time. C. O. C.

Pastes for Coating, Printing, Finishing and Imparting Water-repellency. FBy. *BP* 723,767

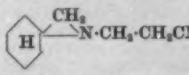
An aqueous solution of an organic binder of mol. wt. > 200 and containing several > NH or $-NH_2$ groups, e.g. a polyvinyl amine, a cross-linking agent containing several radicals capable of reacting with NH or NH_2 groups, e.g. addition product of 1 mol. of a diisocyanate and 2 mol. of a 1:3-diketone, a pigment and if necessary a thickening agent is used. The print or finish and the like being fixed by heat or treatment with an alkali. C. O. C.

Antistatic Agents for Vinyl Resins. American Cyanamid Co. *BP* 723,580

Compounds of formula $R^1{\cdot}CO{\cdot}NH{\cdot}CH_2{\cdot}CH_2NR^2R^3R^4Y$ (R^1 = aliphatic or alicyclic; R^2 and R^3 = Alk or hydroxyalkyl; R^4 = Alk, hydroxyalkyl, alkenyl or aralkyl; Y = anion), e.g. caprylylamidopropyl dimethyl-β'-hydroxyethyl ammonium chloride, are good antistatic agents for use on vinyl resins, 0·2–10·0% on the dry wt. of the resin being used. C. O. C.

Gas Fume Fading Inhibitors. BASF. *BP* 725,353

Fully saturated, 3–7 membered mononitrogen heterocyclic compounds or amines containing *cyclo*aliphatic radicals, having at least one cyanoalkyl group of 1–10 C attached to N, are highly effective gas fume fading inhibitors, e.g. *N*-ω-cyanoethylspirocyclohexylethyleneimine,



gives good protection to cellulose acetate dyed with 1:4-di(monoethylamino)-anthraquinone. C. O. C.

Polymerised Ethyleneimine as a Gas Fume Fading Inhibitor. Celanese Corpn. of America. *BP* 725,678

Cellulose acetate or the like dyed with dyes susceptible to gas fume fading is given excellent protection by incorporation of 0·25–2·0% by wt. of polymerised ethyleneimine of mol. wt. > 550. C. O. C.

Aqueous Suspensions of Halogenomethylated Phosphates as Flame Resisting Agents. U.S. Secretary of Agriculture. *USP* 2,686,768

Flame-resisting agents are produced by heating an aqueous dispersion of a polymerisable, unsaturated alcohol ester of phosphoric acid, e.g. triallyl phosphate, and compound of formula CX_4Y_2 (X = Br or Cl; Y = H, Br or Cl), e.g. CBr_2Cl_2, CBr_4 or $CHBr_3$, in presence of a peroxidic polymerisation catalyst, e.g. K persulphate, to < 200°C. until the non-aqueous phase contains a dispersed polymer containing phosphate and halogenomethyl groups. This yields an acidic dispersion which is neutralised with pyridine. Cellulosic material impregnated with such a dispersion and heated at 110°C. for 30 min. was given a flame-resisting finish which was unimpaired after 8 washings.

USP 2,686,769

An alkali metal bicarbonate (5–8% on the wt. of halogenomethane and phosphate) is added to the aqueous dispersion before polymerisation. This enables a much stronger dispersion to be produced without concentration. Cellulosic material is impregnated with the dispersion so as to take up 25–30% of its dry weight of the polymer and then heated at 100–140°C. for 30–5 min. C. O. C.

Surface-active Agents, Emulsifying and/or Stabilising Agents for Water-in-Oil Emulsions. Unilever. *BP* 723,244

An aliphatic carboxylic acid of > 7C is intermolecularly esterified, i.e. 2 or more molecules are condensed together with elimination of water. The product is then treated with enough of the polycondensate of an aliphatic polyvalent alcohol of > 2 C and > 2 OH, so as to form a partial ester. C. O. C.

***N*-Vinylimidazole-Carboxylic Acid Copolymers—Protective Colloids, Thickening or Dispersing Agent.** BASF. *BP* 726,454

The copolymers of *N*-vinylimidazoles and ethylenically unsaturated aliphatic mono- or dicarboxylic acids of 3–4 C, e.g. *N*-vinyliminazole and maleic acid, have amphoteric properties and form sodium salts which yield viscous aqueous solutions useful as protective colloids, thickening agents and dispersing agents. C. O. C.

Water-repellent Finish. S. C. Johnson & Son. *USP* 2,670,303

A solution containing 1–10% of petroleum waxes and/or petrolatums and/or polymerised octadecyl vinyl ether and an aluminium and/or zirconium alkoxide of 3–18 C imparts optimum water-repellency to wool fabrics and when applied to cotton is resistant to dry cleaning. C. O. C.

Foils and Coatings from Polyvinyl Chloride Pastes. BASF. *BP* 725,705

Foils and coatings having a dull, dry surface and a dry, leather-like handle are obtained by adding to the paste of polyvinylchloride and plasticiser 1–10% by weight of paraffin wax, the wax being added as a 20–60% aqueous emulsion. C. O. C.

Synthetic Tanning Agents. L. Bolgar. *BP* 722,906

An aromatic sulphonic acid is condensed with the product obtained by condensing a ketone with urea in presence of HCHO. C. O. C.

Tanning Agents. Gy. *BP* 723,628

Tanning agents which yield completely white leather of very good light fastness and an even better fullness than that produced by the agents mentioned in *BP* 683,984 (*J.S.D.C.*, **69**, 70 (Feb. 1953)) are obtained by reacting HCHO in aqueous alkaline medium at raised temperature with a mixture of hydroxypolyacylsulphone sulphonic acids and hydroxypolyaryl sulphones. C. O. C.

Organic Polysiloxane Resins for Rendering Textiles Water-repellent (X p. 271).

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

World Production, Export, and Consumption of Colouring Matters. E.-A. Sack. *Teintex*, **20**, 5–11 (Jan. 1955).

Six tables. The market increasingly demands faster colouring matters appropriate to the end use and durability of the substrate. S. R. C.

Progress in Dye Manufacture and in Dyeing, Printing, and Finishing in the U.S.S.R. C. Weimann. *Teintex*, **20**, 103–110 (Feb. 1955).

A review of progress made in 1954. S. R. C.

Auguste Scheurer-Kestner, 1833–1899. E. Justin-Mueller. *Teintex*, **20**, 31–32 (Jan. 1955).

A short biography of the first manufacturer of Guignet's Green. S. R. C.

Observations concerning the Theory of Aromatic Substitution. P. B. D. de la Mare. *J.C.S.*, 4450–4454 (Dec. 1954).

Phenol Sorption on Ion-exchange Resins. R. E. Anderson and R. D. Hansen. *Ind. Eng. Chem.*, **47**, 71–75 (Jan. 1955).

Quaternary ammonium resins can remove phenols from solution in two ways—(*a*) by exchange and sorption on the hydroxide form of the resin, (*b*) by sorption on a salt form of the resin. Each method has advantages and limitations—(*a*) is more suitable for recovering phenols from solutions relatively free from electrolytes, and is suggested as a suitable means of removing phenols from waste effluents, regeneration being by caustic methanol; (*b*) is more suitable for moderate phenol concentrations, particularly in the presence of acids and salts; methanol is a suitable stripping solvent. W. K. R.

Separation of Xylenes—Selective Solid Compound Formation with Carbon Tetrachloride. C. J. Egan and R. V. Luthy. *Ind. Eng. Chem.*, **47**, 250–253 (Feb. 1955).

Carbon tetrachloride forms a solid equimolecular compound with *p*-xylene but not with *m*-xylene or *o*-xylene. By this means, the limitations on *p*-xylene recovery due to eutectic formation with *m*-xylene can be overcome and a substantially increased yield of *p*-xylene can be obtained. After separation of the *p*-xylene, the *m*-xylene is recovered by fractional crystallisation of the carbon-tetrachloride-free mother liquor. W. K. R.

Aromatic Aldehydes from Hydrocarbons via Carboxylic *N*-Methylanilides—VI. F. Weygand and R. Mitgau. *Chem. Ber.*, **88**, 301–308 (Feb. 1955).

Aromatic hydrocarbons and phenol ethers react with methylphenylcarbamyl chloride and aluminium chloride to form aromatic carboxylic *N*-methylanilides, which can be reduced to aldehydes with $LiAlH_4$. In 15 examples cited, yields are 37–84%. H. E. N.

Oxidations with Phenyl Iodosoacetate. IV—Oxidation of Substituted *o*-Nitroanilines. K. H. Pausacker and J. G. Scroggie. *J.C.S.*, 4499–4502 (Dec. 1954).

5-Methyl-, 5-chloro-, and 5-methoxy-2-nitroaniline are oxidised by phenyl iodosoacetate to the corresponding benzofurazan oxides in yields of 90–93% (cf. *J.C.S.*, 1989 (1953)). The products are identical with those obtained by the oxidation of either the 4- or the 5-substituted 2-nitroanilines with alkaline NaClO by Green and Rowe's method (cf. *J.C.S.*, **101**, 2452 (1912)). 1-Nitro-2-naphthylamine under the same treatment gives a mixture of 1:1′-dinitro-2:2′-azonaphthalene (3%) and naphthofurazan oxide (86%), while 2-nitro-1-naphthylamine affords 2-2′-dinitro-1:1′-azonaphthalene (3%) and the same naphthofurazan oxide (7%). From 4-methoxy-2-nitroaniline only 4:4′-dimethoxy-2:2′-dinitroazobenzene could be isolated, but the corresponding benzofurazan oxide was given in quant. yield by alkaline NaClO. The mechanism of the reaction with phenyl iodosoacetate is discussed. H. H. H.

Oxidations with Phenyl Iodosoacetate. V—Oxidation of *p*-Anisidine and *p*-Phenetidine. J. Mitchell and K. H. Pausacker. *J.C.S.*, 4502–4505 (Dec. 1954).

These oxidations yield the 4:4′-dialkoxyazobenzene (in the case of *p*-anisidine), the *NN′*-di-(4-alkoxyphenyl)-*p*-phenylenediamine, the benzoquinone di-*p*-alkoxylphenylimine, the tetra-*p*-alkoxyazophenine, and a cpd. believed to be an azo-subst. diphenylamine. The course of the reaction is discussed. H. H. H.

2:3-Derivatives of Naphthalene. I—Nitration of *N*-Acetyl-3-nitro-1-naphthylamine. E. R. Ward, T. M. Coulson, and J. G. Hawkins. *J.C.S.*, 4541–4545 (Dec. 1954).

Fuming HNO_3 nitrates *N*-acetyl-3-nitro-1-naphthylamine to give a mixture of the 3:5- and 3:8-dinitro deriv., not the 2:3-dinitroamide as claimed by Hodgson and Turner (*J.C.S.*, 635 (1943)). Reduction of 1:3-dinitronaphthalene by catalytic transfer hydrogenation affords a mixture of 3-nitro-1- and 4-nitro-2-naphthylamine. H. H. H.

2:3-Derivatives of Naphthalene. II—Preparation of 2:3-Dinitronaphthalene and 3-Nitro-2-naphthylamine. E. R. Ward and T. M. Coulson. *J.C.S.*, 4545–4547 (Dec. 1954).

An improved synthesis of 2:3-dinitronaphthalene from 1:2:3:4-tetrahydro-6:7-dinitronaphthalene is described, together with its partial reduction to 3-nitro-2-naphthylamine by the procedure of Hodgson and Ward (*J.C.S.*, 1187 (1949)). H. H. H.

Nuclear Substitution by Anions and Self-union in 1:8-Naphthalimide and its *N*-Methyl Derivative. W. Bradley and F. W. Pexton. *J.C.S.*, 4432–4435 (Dec. 1954).

1:8-Naphthalimide is hydroxylated *para* to a CO group with KOH and MnO_2, and sodioaniline converts *N*-methyl-1:8-naphthalimide into the 4-anilino deriv. Neither of the imides is as reactive as *meso*benzanthrone towards these reagents. The self-union of *N*-methyl-1:8-naphthalimide on treatment with alkalis involves the formation of a dinaphthyl deriv. at the first stage, and the cyclisation of this to a perylene deriv. The second stage is facilitated by reducing agents. H. H. H.

Proximity Effects in the Interpretation of the Basic Strengths of Primary Aromatic Amines. R. N. Beale. *J.C.S.*, 4494–4499 (Dec. 1954).

The basic strengths of *o*-, *m*-, and *p*-toluidine, 2:3-, 2:4-, 2:5-, 2:6-, 3:4-, and 3:5-xylidine, and mesidine are determined by a combination of ultraviolet spectrophotometry and pH measurements, and the values are compared with those obtained by other methods. The substituent effect is additive except when two substituents, or a substituent and the basic group, are vicinal. Explanations of the departure from additivity are attempted in terms of electromeric, inductive, and spatial effects. Steric hindrance involving the amino group is absent, and steric strain in, and steric hindrance to solvation of, the anilinium ions appear to play only a very minor rôle in influencing basic strength. In the absence of steric hindrance, the spatial effect of *o*-methyl appears to play an important part in the "*ortho*" phenomenon. H. H. H.

Infrared Absorption Characteristic of Diazonium Cations. M. Aroney, R. J. W. Le Fèvre, and R. L. Werner. *J.C.S.*, 276 (Jan. 1955).

Infrared absorption data are recorded for 21 substances which (apart from the zwitterionic *p*-diazobenzenesulphonic acid) are either diazonium salts with single anions or complex salts containing diazonium cations. The figures are centred around 2261 cm.$^{-1}$ (with standard deviation $\pm$ 21 cm.$^{-1}$), which is of the order to be expected for a triple linkage and thus provides physical evidence for the modern equiv. of Blomstrand's formulation of diazonium salts. The spectra are clear of other group effects, and the occurrence of such absorption may reasonably be accepted for the recognition of the $(R{\cdot}N_2)^+$ cation. H. H. H.

Mechanism of Diazotisation. E. Abel. *Mh. Chem.*, **85**, 1169–1171 (Oct.); 1312–1314 (Dec. 1954).

I—The possibility is discussed of deciding experimentally between the mechanism for diazotisation proposed by Hughes, Ingold *et al.*, and by Schmid and co-workers, which involves the step—

$$2\,HNO_2 \rightarrow N_2O_3 + H_2O$$

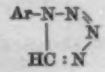
and that proposed by Abel *et al.*, which supposes that—

$$2\,HNO_2 \rightarrow NO_2 + NO + H_2O.$$

II—Employing the kinetics of the oxidation of nitrous to nitric acid determined by Reinders and Vles (*Rec. Trav. chim.*, **44**, 1 (1925)), a decision in favour of the former mechanism is reached. H. E. N.

Coupling of Aromatic with Aliphatic Diazo Compounds. R. Huisgen and H.-J. Koch. *Annalen*, **591**, 200–231 (Feb. 1955).

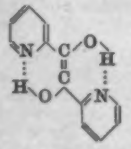
On coupling aromatic diazo cpd. with various types of aliphatic diazo cpd. products of the following structures are obtained: with ethyl diazoacetate Ar·NH·N:CCl·COOC$_2$H$_5$; with diazoketones Ar·NH·N:CCl·CO·R; with diarylidiazomethanes (in methanol) Ar^1·N:N·C(O·CH$_3$)·Ar^2; with diazomethane Ar·NH·N:CHCl, but—



Ar·N(CH$_3$)·C:N, and other cpd. can also be obtained; and with diazoethane Ar·NH·N:CCl·CH$_3$. H. E. N.

Reaction of Diazonium Salts with α-Pyridoin. B. Eistert and H. Munder. *Chem. Ber.*, **88**, 226–233 (Feb. 1955).

Aq. benzenediazonium sulphate reacts with an aq. acetone soln. of α-pyridoin—



to give α-picolinic acid and its phenylhydrazide in > 53% yield. The mechanism of this reaction is investigated and discussed. H. E. N.

Photochemical Decomposition of Hydrazobenzene, studied by the use of Nitrogen Isotopes. P. F. Holt and B. P. Hughes. *J.C.S.*, 98–100 (Jan. 1955).

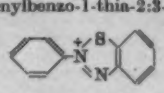
Photolysis of [^{15}N$_1$]hydrazobenzene produces azobenzene which has the same isotope distribution as the starting material. Possible mechanisms of the reaction are discussed. The syntheses of [^{15}N$_1$]hydrazobenzene and [^{15}N$_1$]benzidine are described. H. H. H.

Infrared Spectra of some Unsulphonated Monoazo Dyes. M. Dolinsky and J. H. Jones. *J. Assocn. Off. Agric. Chem.*, **37**, 197–209 (Feb. 1954).

The infrared spectra of a number of unsulphonated azo dyes and related compounds are given. The nature of the absorption curves and their value in establishing the location and nature of substituents in the azo dye molecule are discussed. P. G. M.

***o*-Mercapto-azo Compounds. VI—Preparation and Structure of Azobenzene-2-sulphenyl Derivatives.** A. Burawoy, F. Liversedge, C. E. Vellins, G. W. R. Bartindale, and G. Farrow. *J.C.S.*, 4481–4486 (Dec. 1954).

The preparations are described of azobenzene-2-sulphenyl cyanide, chloride, iodide, thiocyanate, and perchlorate from the bromide (cf. *J.C.S.*, 90 (1954)). Various physical data are recorded, e.g. equiv. conductivities, mol. wt. in H_2O and $CHBr_3$, and electronic spectra in different solvents, and show that with the exception of the non-ionic cyanide, all the azobenzene-2-sulphenyl deriv. exist in H_2O and C_2H_5OH as true salts involving a 2-phenylbenzo-1-thia-2:3-diazolium ion—



and in C_6H_6 and $CHCl_3$ as equilibrium mixtures of the ionic and non-ionic isomers. The ionic structure of cryst. azobenzene-2-sulphenyl chloride is established by X-ray analysis. H. H. H.

Formation of Carbonium Ions by the Action of Metal Salts. I—Reaction of Mercuric Chloride with Triphenylmethyl Chloride in Nitromethane. J. W. Bayles, A. G. Evans, and J. R. Jones. *J.C.S.*, 206–211 (Jan. 1955).

The following equilibria exist, involving triphenylmethyl ions, in the reaction between $HgCl_2$ and $(C_6H_5)_3CCl$ in nitromethane—

$$(C_6H_5)_3CCl + HgCl_2 \rightleftharpoons (C_6H_5)_3C^+HgCl_3^-$$

$$\text{and } (C_6H_5)_3C^+HgCl_3^- \rightleftharpoons (C_6H_5)_3C^+ + HgCl_3^-$$

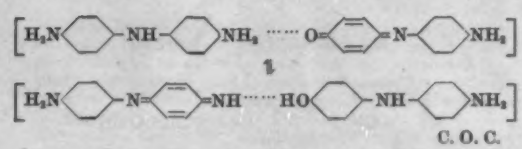
The concn. of the ions has been measured spectrophotometrically, and the changes in free energy and heat content for the forward reactions have been determined. H. H. H.

Azomethin and Formazyl Complexes. M. Seyhan. *Chem. Ber.*, **88**, 212–215 (Feb. 1955).

The anil from 5:6-benzoquinoline-2-aldehyde (I) and *o*-aminophenol reacts spontaneously with uranyl acetate in alcoholic soln. to form a red complex. The formazan obtained by coupling diazotised anthranilic acid with the phenylhydrazone of I similarly yields a dark red complex. The formazan obtained using diazobenzene did not form a uranium complex. H. E. N.

Constitution of Phenylene Blue. K. W. Merz and A. Kammerer. *Arch. Pharm.*, **286**, 139–150 (1953): *Chem. Abs.*, **49**, 926 (25 Jan. 1955).

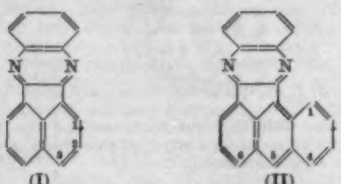
The colour base of Phenylene Blue, almost black microcrystals, has been isolated for the first time in 2·65–3·0 g. yields by adding 12 g. finely powdered $K_2S_2O_8$ with shaking to 4 g. aniline hydrochloride and 5·6 g. *p*-diaminobenzene hydrochloride in 300 ml. water, adding 700 ml. $CHCl_3$ and 300 ml. 5·3% aq. NaOH after 1–2 min. shaking, removing the $CHCl_3$ layer, extracting twice more with 300 ml. and 200 ml. $CHCl_3$, washing the combined $CHCl_3$ extracts with 400 ml. aq. NH_4OH, filtering at once, drying with anhydrous Na_2SO_4, evaporating *in vacuo* under N to 250 ml., adding 1200 ml. petroleum ether, and allowing to stand in the cold 2–3 days. Phenylene Blue is only difficultly soluble in ether, benzene, $CHCl_3$ and CCl_4 to red solutions. It is fairly soluble in acetone, dioxan, methyl and ethyl alcohols, the first two yielding red and the alcohols violet solutions. With dilute acids solutions of the blue colour salt are obtained. On catalytic hydrogenation of I in absolute alcohol or dioxane using Pd-$BaSO_4$ or Pd-C, 0·5 mole H was taken up. Reduction of Phenylene Blue with C_6H_5·$NHNH_2$ yields 0·5 mole N. When the hydrogenated product was treated with C_6H_5·$NHNH_2$ no N was evolved. Therefore Phenylene Blue cannot be *p*-aminophenyl-*p*-benzoquinone dimer. It is probably a quinhydronelike addition compound of *pp'*-diaminodiphenylamine and *N*-*p*-aminophenyl-*p*-benzoquinone imine, and, respectively, of *N*-*p*-aminophenyl-*p*-benzoquinone diimine and *p*-hydroxy-*p'*-aminodiphenylamine—



C. O. C.

Self-union Reactions and Nuclear Substitution by Anions in Some Ring Homologues of Quinoxaline. W. Bradley and F. W. Pexton. *J.C.S.*, 4436–4439 (Dec. 1954).

Acenaphtho[1,2-*b*]quinoxaline (I) affords a small amount of the 3-hydroxy deriv. when heated with KOH, whereas acenanthra[1,2-*b*]quinoxaline (II) does not. Both I and II undergo self-union to deriv. of perylene when heated with KOH. The relation of these reactions to those of related carbonyl cpd. is discussed.



(I) (II)

H. H. H.

Colour of Quinones. E. Cerutti and J. Martinet. *Compt. rend.*, 237, 1336–1338 (1953): *Chem. Abs.*, 49, 996 (25 Jan. 1955).

Two rules enable the colour of quinones to be deduced from that of the parent hydrocarbons. (1) The longer the conjugation between the two carbonyls the deeper the colour (*o*-quinones, however, have more colour than *p*-quinones; this may be due to conjugation between two COs being better than that between CO and C:C). (2) Influence of relative number of Kekulé forms in hydrocarbon and quinone. If N_q and N_h = number of Kekulé forms in the quinone and the hydrocarbon, respectively, then $N_h - N_q/N_h + N_q = R$. When $R > 0$, the quinone is more coloured than the parent hydrocarbon; when $R = 0$, quinone and hydrocarbon are about the same colour; when $R < 0$, the hydrocarbon is more deeply coloured than the quinone, e.g. benzene—colourless, *p*-benzoquinone—yellow ($R = 0{\cdot}33$); 1:2:6:7-dibenzoanthraquinone—yellow, parent hydrocarbon—yellow ($R = 0$); 6:13-pentacenequinone—yellow; pentacene—bluish violet ($R = -0{\cdot}2$). When R is large, oscillations between 2 O atoms are more favoured than between two C atoms and thus the quinone is more deeply coloured than the hydrocarbons. The unshared electrons around the O atoms also act as auxochromes by contributing to the oscillating system. C. O. C.

Disperse Dyes. II—Synthesis of 1:4-Disubstituted 2-Hydroxyethylaminoanthraquinones. Y. Bansho. *J. Chem. Soc. Japan, Ind. Chem. Sectn.*, 57, 80–83 (1954): *Chem. Abs.*, 49, 1329 (25 Jan. 1955).

Quinizarin (100 g.), recrystallised from chlorobenzene, dispersed in water (2 l.) and soda ash (100 g.), was stirred with hydrosulphite (60% purity, 200 g.) at 80°C. for 1 hr. in an atmosphere of N to yield leucoquinizarin (96·5 g.). Leucoquinizarin (4·84 g.) dispersed in water (24 c.c.) and monoethanolamine (7·2 g.) and then boiled for 8 hr. yielded green crystals of 1:4-bis(2-hydroxyethylamino)-anthraquinone (0·08 g.) (II). Treating II with $SOCl_2$ at 140–150°C. for 15 min. in nitrobenzene yielded blue-violet crystals. 1:4:5:8-Tetraaminoanthraquinone (III) (15 g.) was treated with hydrosulphite (60% purity) (37·5 g.) in water (320 c.c.) and NaOH (27 g.) at 70–120°C. for 2–3 hr. to yield the dark brown leuco-III (14 g.). Leuco-III (4 g.) was added to water (30 c.c.) and alcohol (3 c.c.) and treated with hydrosulphite (1 g.) at 60°C. for 3 hr. to yield a compound (4·2 g.) which gave a reddish brown solution in butyl alcohol. Heated at 130°C. for 18 min. in nitrobenzene (10 g.) and $SOCl_2$ (0·1 c.c.) this compound yielded 1:4-bis(2-hydroxyethylamino)-5:8-dihydroxy-anthraquinone (3·58 g.), a green-blue dye, similar to Celliton Fast Blue-Green B. C. O. C.

Synthesis of Benzanthrone and Dyes derived from it. III—Synthesis of *iso*Dibenzopyrenequinone from Naphthalene. E. Koike, M. Okawa, and T. Kaneko. *J. Chem. Soc. Japan, Ind. Chem. Sectn.*, 55, 535–538 (1952): *Chem. Abs.*, 49, 993 (25 Jan. 1955).

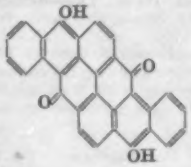
1-Benzoyl-4-benzylnaphthalene (I), 79%, was obtained by condensing *a*-benzylnaphthalene with benzoyl chloride in presence of $AlCl_3$ at 30°C. 1:4-Dibenzoylnaphthalene (II), 80·9%, was obtained by boiling I with 20% HNO_3 for 10 hr. *iso*Benzopyrenequinone (III), 67·8%, was obtained by fusing II with a mixture of $AlCl_3$ and NaCl ($AlCl_3$:NaCl::7:1 by wt.) at 145°C. for 30 hr. 1:4-Dibenzylnaphthalene (IV), 51·3%, was obtained by treating naphthalene with $C_6H_5{\cdot}CH_2Cl$. III, 56·9%, was obtained by boiling IV with 20% HNO_3. C. O. C.

Direct Hydroxylation of 6-Phenyl*meso*benzanthrone and Two Benzo Derivatives of *meso*Benzanthrone, and some Related Reactions. A. J. Backhouse, W. Bradley, and F. K. Sutcliffe. *J.C.S.*, 91–95 (Jan. 1955).

When the 6-phenyl, and the 5:6- and 8:9-benzo-*meso*-benzanthrones are heated with KOH, direct hydroxylation in the 4-position occurs; the reaction is facilitated by addition of MnO_2. With the first cpd., sodioaniline affords the 4-anilino deriv. 3-Bromo*meso*benzanthrone yields the 6-phenyl deriv. with phenylmagnesium bromide, an example of the rule that nuclear substitution is the predominant reaction when the reacting anion is highly active. The formation of the monohydroxy deriv. above supports the conclusion (cf. *J.C.S.*, 2118 (1951)) that the number of OH groups introduced by KOH fusion is equal to the number of activating CO groups present. H. H. H.

Direct Hydroxylation of Pyranthrone and *amphi-iso*Pyranthrone. A. J. Backhouse and W. Bradley. *J.C.S.*, 4506–4507 (Dec. 1954).

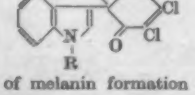
The title cpd. yield the same dihydroxy deriv., viz. 3:11-dihydro-8:16-dihydroxy-3:11-dioxopyranthrene (I) when heated with KOH.



H. H. H.

o-Quinones. VI—Reaction with Tertiary Amines and its relation to Melanin Formation. L. Horner and W. Spietschka. *Annalen*, 591, 1–20 (Feb. 1955).

Tetrachlorobenzo-*o*-quinone forms quaternary ammonium salts with aromatic tertiary bases having free *ortho* positions, such as pyridine, *iso*quinoline, and *γ*-lepidine, but with *α*-picoline, collidine, quinoline, quinaldine, and lepidine quinhydrone-like cpd. are formed. With aliphatic amines, such as triethylamine, dimethyl- and diethyl-aniline, and *N*-ethylpiperidine explosive oxidation of an alkyl group occurs. Indole and its *N*-alkyl deriv. react to form orange–yellow cpd., which are not quinhydrones, via deeply coloured quinhydrones. The former are shown to have the structure—



The mechanism of melanin formation is discussed. H. E. N.

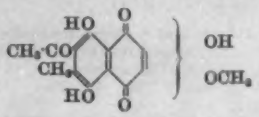
Synthesis of Organic Fluorescent Compounds. XII—Emissivity of Fluorescence of Naphth-[1,2]iminazoles and Perimidines. Synthesis of Perimidines. Z. Yoshida, Y. Shimada, and R. Oda. *J. Chem. Soc. Japan, Ind. Chem. Sectn.*, 55, 523–524 (1952): *Chem. Abs.*, 49, 1021 (25 Jan. 1955).

Treating 1:8-diaminonaphthalene with various acids and acid chlorides yielded the following perimidines—

perimidine (47%), 2-methylperimidine (54%), 2-phenylperimidine (50%), 1:4-bis(2-perimidyl)benzene (28·5%), 1:5-bis(2-perimidyl)naphthalene (48%). Formation of perimidines is easier than that of naphth[1,2]iminazoles, i.e. amino groups in the 1:8 positions in naphthalene are more active than those in the 1:2 positions. C. O. C.

Cordeauxiaquinone, Colouring Matter of the Leaves of *Cordeaux edulis* (Hemsl.). J. H. Lister, C. H. Eugster, and P. Karrer. *Helv. Chim. Acta*, **38**, 215–222 (Feb. 1955).

This shrub grows in Ethiopia and Somaliland. To the only colouring matter, a dark red cpd., m.p. 194°c., which could be isolated from the leaves, is assigned the formula—



H. E. N.

***mero*Cyanines derived from Thio-oxindole. III—Trinuclear *mero*Cyanines.** R. H. Glauert, F. G. Mann, and A. J. Wilkinson. *J.C.S.*, 28–30 (Jan. 1955).

The following four trinuclear *mero*cyanines containing the thio-oxindole nucleus are prepared and described—3-ethyl-5-(3-ethylbenzothiazolin-2-ylidene-ethylidene)-2-(2:3-dihydro-2-oxothionaphthen-3-ylidene)thiazolid-4-one, 4-(3-ethylbenzothiazolin-2-ylidene-ethylidene)-2-(2:3-dihydro-2-oxothionaphthen-3-ylidene)-3-methylthiazolid-5-one, 3-ethyl-5-(3-ethylbenzothiazolin-2-ylidene-ethylidene)-2-(2:3-dihydro-3-oxothionaphthen-2-ylidene)-thiazolid-4-one, and 4-(3-ethylbenzothiazolin-2-ylidene-ethylidene)-2-(2:3-dihydro-3-oxothionaphthen-2-ylidene)-3-methylthiazolid-5-one. These dyes are compared briefly with their isomers derived from thioindoxyl.

H. H. H.

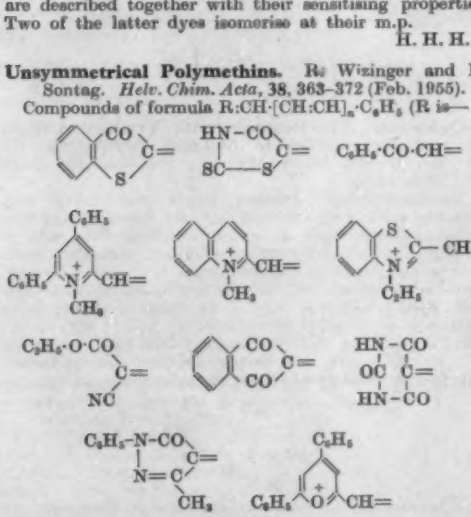
Oxonols derived from Thio-oxindole—I. R. H. Glauert, F. G. Mann, and A. J. Wilkinson. *J.C.S.*, 30–34 (Jan. 1955).

The symmetrical oxonol derived from thio-oxindole and three unsymmetrical oxonols containing this nucleus are described together with their sensitising properties. Two of the latter dyes isomerise at their m.p.

H. H. H.

Unsymmetrical Polymethins. R. Wizinger and H. Sontag. *Helv. Chim. Acta*, **38**, 363–372 (Feb. 1955).

Compounds of formula $R{:}CH{\cdot}[CH{:}CH]_n{\cdot}C_6H_5$ (R is—

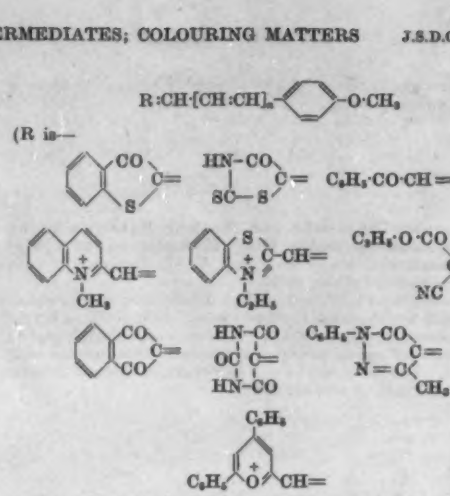


and $n = 0$–5) are prepared and their absorption maxima determined. The difference, λ_{max} for $n = 5$ minus λ_{max} for $n = 0$, increases in the order given. The shifts in λ_{max} as n increases are tabulated and discussed.

H. E. N.

Unsymmetrical Polymethins. R. Wizinger and P. Kölliker. *Helv. Chim. Acta*, **38**, 372–380 (Feb. 1955).

Compounds of formula—



and $n = 0$, 1, or, in the last three cases only, 5) are prepared and their absorption maxima determined. By comparison with the cpd. not carrying the methoxyl group, the bathochromic effect of this group is shown to diminish as n increases. H. E. N.

Absorption Spectra and Chemical Structure. I—Conjugated Polyenes and *p*-Polyphenyls. II—Solvent Effect. III—Effects of Substituents and Structure. IV—Unsaturated Aldehydes, Ketones, and Carboxylic Acids. K. Hirayama. *J. Amer. Chem. Soc.*, **77**, 373–384 (20 Jan. 1955).

Study of the dependence of spectral absorption in polyene derivatives and *p*-polyphenyls has shown that there is a quantitative relationship between λ_{max} (in the long-wave absorption band) and the molecular structure of certain polyenes, and that the same relationship holds good for conjugated systems having either a carbonyl or a carboxyl group in α-position or in both the α- and ω-positions. Allowance must be made for the solvent. The effects of furanoid oxide substituents and of certain spectroscopically effective rings are discussed. C. O. C.

Synthesis of Carotenoids. R. Ahmad. *J. Imp. Coll. Chem. Soc.*, **31**, 23–37 (1953): *Chem. Abs.*, **49**, 967 (25 Jan. 1955).

A review of the complete synthesis of β-carotene, 35 references. C. O. C.

Proton-acceptor Properties of Carotene. A. Wassermann. *J.C.S.*, 4329–4336 (Dec. 1954).

Spectrophotometric experiments are described which deal with proton-acceptor properties of carotene, a deeply coloured hydrocarbon containing 10 conjugated double bonds in the α-modification and 11 in the β-form. The basicity of these polyenes is sufficiently large for proton-transfer reactions to be studied in an aprotic solvent. A proton-transfer mechanism proposed for the formation of these adducts is found to be consistent with a relation of the Brønsted type between K and the dissociation constants of the three chloroacetic acids, and with the fact that addition of acids to carotene is prevented by basic solvents. H. H. H.

Pyrrole Dyes. H. Zinner. *Urania* (Germany), **15**, 65–70 (1952): *Chem. Abs.*, **49**, 1116 (25 Jan. 1955).

A discussion of the chemistry of pyrrole, porphyrin, haemin, chlorohaemin, haemoglobin, biliverdin, chlorophyll, xanthophyll, carotenes, and metallophthalocyanines. C. O. C.

Ion Exchange in Ultramarine. R. M. Barrer and J. S. Raitt. *J.C.S.*, 4641–4651 (Dec. 1954).

Ions which exchange fairly readily in ultramarine include Na^+, K^+, Li^+, Ag^+, Tl^+, Pb^{++}, Zn^{++}, and Cd^{++}, while those which do not exchange readily include Ca^{++}, Sr^{++}, Ba^{++}, Mg^{++}, NH_4^+, Rb^+, and Cs^+. Factors such as ion size, valency, basigenic character, polarisability, and tendency to form covalent links and insol. sulphides, all appear to be involved in determining the relative affinity of ions for the crystal. The instability of ultramarine under hydrothermal conditions reduces its value as a

medium for the quant. study of ion exchange. Ion exchange can proceed smoothly when various dry org. solvents, e.g. benzene, acetone, and *sec.*-butyl and *iso*-pentyl alcohols, are used in place of water. Ion-exchange diffusion coefficients, energies, free energies, and entropies of activation are recorded for $Na^+ \rightarrow Ag^+$, $Ag^+ \rightarrow Na^+$, $Ag^+ \rightarrow Li^+$, $Ag^+ \rightarrow K^+$, and $Na^+ \rightarrow Tl^+$. X-Ray powder photographs of the ion-exchange products indicate the ultramarine structure to show slight adjustments in lattice dimensions when different cations are introduced. Evidence of limited mutual solid solubility of end-members of an exchange pair was found only with Ag and Zn and with Ag and K ultramarines. H. H. H.

PATENTS

α-Amino-β-bromoanthraquinones. General Aniline. *USP* 2,650,928

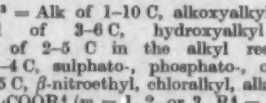
α-Aminoanthraquinones are brominated in a melt of $AlCl_3$ and NaCl containing some tertiary amine, e.g. pyridine to give pure β-bromo derivatives. Thus, all parts being by weight, 1-amino-2-bromoanthraquinone is prepared by adding bromine (75) over 90–120 min. to 1-aminoanthraquinone (112) in a melt of $AlCl_3$ (770), NaCl (115) and pyridine (77) at 85–90°C., and finally stirring at 100°C. for 4 hr. R. K. F.

Metal-complex Monoazo Dyes for Wool and Nylon. Ciba. *BP* 724,157

2-Aminophenol-5- and -6-sulphonamides are diazotised and coupled with 5-chloro- or 5:8-dichloro-1-naphthol, and the monoazo compounds so formed converted into chromium or cobalt complexes containing 1 atom metal to 2 mol. monoazo compound. Alternatively, 1 mol. of the monoazo compound is combined with 1 atom of chromium or cobalt and 1 mol. of a monoazo compound made by coupling a diazotised *o*-aminophenol with 5-chlor- or 5:8-dichloro-1-naphthol or with a 1-acylamino-7-naphthol. The various diazo and coupling components used may contain common substituents, but must be free from SO_3H and COOH groups. The water-soluble metal-complex dyes so formed dye wool and nylon from neutral, weakly alkaline, or weakly acid baths. Thus the monoazo compound 2-aminophenol-5-sulphonamide → 5:8-dichloro-1-naphthol is dissolved in aq. NaOH and treated for ½ hr. at 70–80°C. with cobalt sulphate. The product dyes wool violet. E. S.

Blue Monoazo Dischargeable Dyes for Cellulose Acetate. Eastman Kodak Co. *BP* 723,933

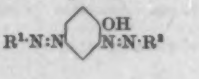
Diazotised 2-amino-5-nitrothiazoles couple with appropriate coupling components to give blue dyes for cellulose acetate of good fastness to light and burnt gas fumes, and readily dischargeable. They are of formula—



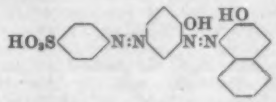
(R^2 and R^3 = Alk of 1–10 C, alkoxyalkyl or hydroxyalkoxyalkyl of 3–6 C, hydroxyalkyl of 2–5 C, cyanoalkyl of 2–5 C in the alkyl residue, sulphoalkyl of 2–4 C, sulphato-, phosphato-, or phosphonoalkyl of 2–5 C, β-nitroethyl, chloralkyl, alkenyl of 2–4 C, or $-(CH_2)_m\cdot COOR^4$ (m = 1, 2, or 3, R^4 = Alk of 1–4 C); R^1 = H, Alk of 1–6 C, CN, CF_3, phenyl, nitro-, halogeno-, or alkyl (1–4 C)- phenyl; Z = Alk of 1–4 C, alkoxy or hydroxyalkoxy of 1–4 C, Hal or –NH·CO·Alk (1–3 C); n = 0, 1 or 2). Thus nitrosyl sulphuric acid is prepared by adding sodium nitrite to conc. H_2SO_4, and a mixture of acetic and propionic acid is added. At 0–5°C. 2-amino-5-nitrothiazole is added followed by more acetic–propionic acid mixture, and urea to destroy excess nitrous acid. The diazo soln. so produced is added to a soln. in acetic-propionic acid mixture of *NN*-bis-β-hydroxyethyl-*m*-toluidine. After adding sodium acetate until neutral to Congo Red, the mixture is diluted with ice water and the dye filtered off. It dyes cellulose acetate bright, deep blues. E. S.

Chromable Disazo Wool Dyes. S. *BP* 723,632

oo'-Dihydroxydisazo compounds



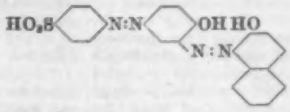
(R^1 = subst. or unsubst. benzene or naphthalene radical; R^2 = radical of *ortho*-coupling naphthol or keto-methylene compound) containing at least one water-solubilising group) are afterchrome or metachrome browns, reds or greens for wool. Three methods of preparation are available, viz. (1) Monoazo compounds $R^1\cdot NH_2 \rightarrow$ *o*-acylaminophenol are de-acylated, and the resulting aminoazo compound diazotised and coupled with HR^2. (2) The nitro group of monoazo compounds $R^1\cdot NH_2 \rightarrow$ *o*-nitrophenol is reduced to NH_2, and the product diazotised and coupled with HR^2. (3) $R^1\cdot NH_2$ is diazotised and coupled with *oo'*-dihydroxymonoazo compounds *o*-aminophenol → R^2. Thus diazotised sulphanilic acid is diazotised and coupled with *o*-hydroxyacetanilide, the product de-acetylated, diazotised and coupled with β-naphthol to give the greyish brown afterchrome or metachrome dye.



E. S.

Metallisable, Disazo Dyes for Wool and Nylon. FBy. *BP* 723,719

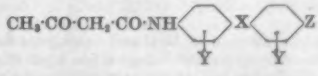
Aminosulphonic acids of benzene or naphthalene are diazotised and coupled with *o*-acylaminophenols, the acyl group is then split off, and the resulting aminomonoazo compound diazotised and coupled with an *ortho* coupling naphthol, pyrazolone or acetoacetarylamide to yield dyes which are converted into chromium complexes in substance on the fibre, or in the dyebath. Thus the acetyl group is split off from the monoazo dye sulphanilic acid → *o*-acetamidophenol by boiling with aq. NaOH. The resulting aminomonoazo compound is diazotised and coupled with a soln. of β-naphthol in aq. NaOH to yield



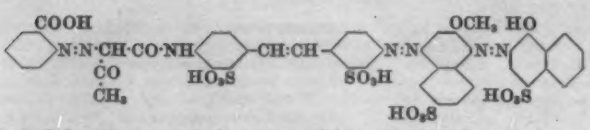
which dyes wool red, converted to black-brown by afterchroming. E. S.

Metal-complex Tris- and Tetrakis-azo Direct Cotton Dyes. S. *BP* 723,637

One mol. of a diazotised *o*-aminocarboxylic acid of the benzene or naphthalene series is coupled with an acetoacetamide derivative



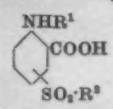
(X = direct link, CH:CH, NH·CO, CO·NH, or NH·CO·NH; Y = H, COOH or SO_3H; Z = NH_2, NO_2 or NH Acyl) and converted if necessary by reduction (if Z = NO_2) or by hydrolysis (if Z = NH Acyl) into the aminoazo compound, which is then diazotised and coupled with a *para*-coupling primary amine of the benzene or naphthalene series which has a group capable of taking part in metal-complex formation *ortho* to its amino group (e.g. *o*-anisidine). The aminodisazo compound so formed is then diazotised and coupled with an *ortho*-coupling naphthol which may carry substituents such as OH, NH_2, NH Acyl, N:N-Aryl, or N:N-pyrazolone. The products are metallised, usually with copper salts, to give mainly green to olive direct cotton dyes. Thus anthranilic acid is diazotised and coupled with 4-acetoacetamido-4′-nitrostilbene-2:2′-disulphonic acid, and the nitro group then reduced with Na_2S. The aminoazo compound so formed is diazotised and coupled with 2-methoxy-1-naphthylamine-6-sulphonic acid, and the resulting aminodisazo compound is diazotised and coupled with 1-naphthol-4-sulphonic acid. The trisazo dye so formed, viz.—



is boiled with ammonical copper sulphate to give the copper complex, which dyes cellulose olive. Alternative preparations for making the same dyes are available. E. S.

Azoic Violets, Blues and Greens for Development by Neutral Steaming. FBy. *BP* 723,389

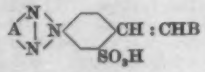
Azoic diazo components used for violets, blues and greens do not combine satisfactorily with the amines—



(R^1 = H or Alk; R_2 = the radical of an aminoalkyl sulphonic or carboxylic acid, or Alk containing a solubilizing substituent) of *BP* 715,249 (*J.S.D.C.*, **70**, 522 (1954)) to give diazoamino compounds, but their diazo compounds may be combined with other suitable stabilizers consisting of secondary amines containing solubilizing groups, e.g. sarcosine, methyl taurine, cyclohexylaminoacetic acid, proline, and *N*-ethyl-5-sulphoanthranilic acid. E. S.

Fluorescent Benzotriazole Compound—Fluorescent Brightening Agents. Gy. *BP* 726,119

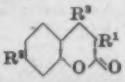
Compounds of formula—



(A = Subst. or unsubst. *o*-phenylene; B = subst. or unsubst. benzene; in either case no substituent may be a chromophor or salt-forming auxochrome) are only slightly coloured, have strong blue fluorescence under ultraviolet radiation and are very stable to chlorine. Thus the sodium salt of 6-methoxy-2-(stilbyl-4′)-1:2:3-benzotriazole-2′-sulphuric acid is yellowish-orange, dyed on cellulose it yields a bluish-white of good fastness, particularly to chlorine. C. O. C.

Fluorescent Brightening Agents for use with Alkaline Detergents. Procter & Gamble Co. *USP* 2,673,186

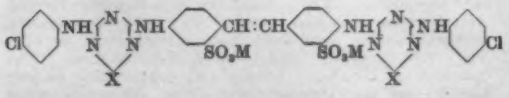
Compounds of formula—



(R^1 = Alk or aralkyl of < 16 C and having a satd. C atom adjacent to the coumarin ring; R^2 = hydroxy, amino, mono- or dialkylamino or heterocyclicamino of < 7 C; R^3 = H or Alk of < 7 C), e.g. 3-*iso*propyl-7-hydroxycoumarin, are blue fluorescing compounds of good stability to alkali. C. O. C.

Fluorescent Brightening Agents of Good Fastness to Chlorine. American Cyanamid Co. *USP* 2,671,784

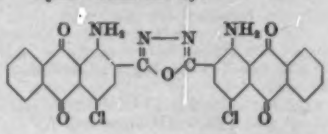
Compounds of formula—



(X = 2-hydroxyethylamino, bis(2-hydroxyethyl)amino or morpholinyl; M = H, NH_4 or an alkali metal) have excellent resistance in aqueous solution to sodium hypochlorite and light. C. O. C.

Halogenated Aminoanthraquinonyl—Oxdiazoles—Vat Dyes. DuP. *USP* 2,650,926

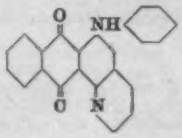
The bis(aminoanthraquinonyl)oxdiazoles of *USP* 2,464,831 are chlorinated, e.g. with *N*:*N*-dichloro-*p*-toluenesulphonamide, or brominated with Br to give 4′-mono- or 4′:4″-dihalogenated derivatives which are red vat dyes. Thus the dye—



is prepared by adding *N*-*N*-dichloro-*p*-toluenesulphonamide to a suspension of 2:5-bis(1′amino-2′-anthraquinonyl)-1:3:4-oxdiazole in nitrobenzene and trichlorobenzene at 175–180°C. R. K. F.

Arylamino – Pyridinoanthraquinones— Disperse Dyes. Celanese Corpn. of America. *USP* 2,658,898

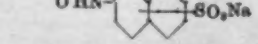
Blue disperse dyes fast to light, washing and burnt gas fumes are made by condensing the leuco derivative of 1-hydroxy-3:4(N)-pyridinoanthraquinone with aniline, or a nuclear mono-substituted aniline. Thus the dye—



is prepared by treating 1-hydroxy-3:4(N)-pyridinoanthraquinone, itself made by a Skraup synthesis from 1-amino-4-hydroxyanthraquinone, with aq. NaOH and sodium hydrosulphite at 60–70°C. for 1 hr., cooling to 20°C., and acidifying with acetic acid. The resulting leuco compound is refluxed for 20 hr. with aniline and boric acid in ethanol, diluted with water and aerated by passing air through the resulting suspension. R. K. F.

Anthraquinone Naphthocarbazole Acid Dyes. Allied Chemical & Dye Corpn. *USP* 2,658,899

Brown acid dyes are prepared by cyclising and simultaneously sulphonating a 1-[alkyl(cycloalkyl or aryl)sulphonylbenzamido]-4-β-naphthylaminoanthraquinone containing a 2-methyl, 2-halogen or 2-sulphonic acid. Thus the dye—



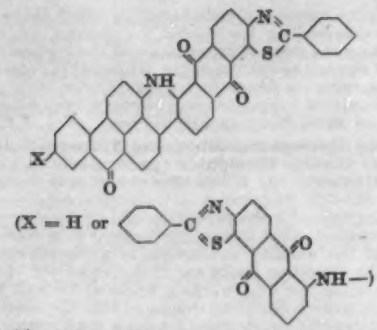
is made by treating 1-[*p*-(methylsulphonyl)-benzamido]-2-methyl-4-(β-naphthylamino)-anthraquinone at 40°C. for 5 hr. with H_2SO_4. Separation is effected by pouring into aq. Na_2SO_4. R. K. F.

Chloromethylated Indigos and "Onium" Salts prepared from them. General Aniline. *USP* 2,657,214

Water-soluble indigo derivatives which may e.g. be printed directly on cotton are prepared by first introducing chloromethyl groups and then converting these into the corresponding methyl *iso*thiouronium and methyl quaternary ammonium salts. Thus indigo is stirred for 2 hr. at 0°C. with bis-chloromethyl ether and H_2SO_4 and poured on to ice. The resulting monochloromethylindigo is heated on a steam bath for 1 hr. with aq. trimethylthiourea and the *iso*thiouronium salt thus formed separated by adding acetone. R. K. F.

Benzanthrone-Acridine Vat Dyes containing a Thiazole Ring. American Cyanamid. *USP* 2,655,502

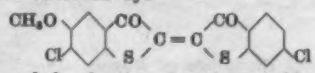
Compounds of formula—



are khaki vat dyes. They are made by first forming the anthrimide by condensing 3-bromo or 3:9-dibromobenzanthrone with 1-amino-5:6-phenylthiazolanthraquinone and cyclising to the acridine. Thus 1-amino-5:6-phenylthiazolanthraquinone, 3 - bromobenzanthrone, sodium and copper acetate are stirred at 200°c. in nitrobenzene. The resulting product is then heated at 120°c. with a mixture of equal parts of ethanol and KOH.
R. K. F.

Asymmetrical Indigoid Dyes. Ciba. *BP* 723,468

A 5-alkoxy-6-chloro-3-oxythionaphthene is condensed with another oxythionaphthene (or its anil derivative), or an isatin, to give a series of light fast indigoid dyes. Thus the violet-red dye—



is prepared by heating 5-methoxy-6-chloro-3-oxythionaphthene (made from 4-methoxy-3-chloro-1-aminobenzene via the thioglycollic acid) together with the 2-(*p*-dimethylamino)-anil of 6-chloro-3-oxythionaphthene in chlorobenzene at 80–90°c.
R. K. F.

Phthalocyanine Process. American Cyanamid. *USP* 2,657,213

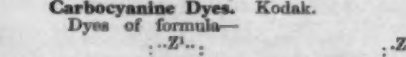
The difficulties arising from loss of phthalic anhydride, choking of condensers, etc., in the preparation of phthalocyanine by the phthalic anhydride–urea–inert solvent method are avoided by including a second inert solvent b.p. < 150°c., e.g. monochlorobenzene, in an amount which is 10–15% by vol. of the first solvent.
R. K. F.

Phthalocyanine Dyes. FBy. *BP* 724,212

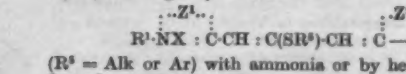
The dyes of *BP* 717,137 (*J.S.D.C.*, **71**, 112 (Feb. 1955)) are mono- and di-methylated on the N of the $-CH_2NH_2$ groups to produce blue dyes for tanned cotton and wool which are redder than those of the unmethylated products. Thus di-*w*-aminomethyl–copper–phthalocyanine is heated for 5 hr. at 90°c. with aq. formaldehyde and formic acid, and the mixture diluted with water and made slightly alkaline with aq. NaOH.
R. K. F.

Carbocyanine Dyes. Kodak. *BP* 726,148

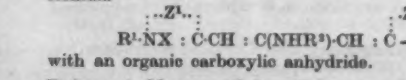
Dyes of formula—



(R^1 and R^4 = same or different, subst. or unsubst. Alk; R^2 = H, subst. or unsubst. Alk or aromatic carbocyclic group; R^3 = acyl or where R^2 = H may also = H; X = anion; Z^1 and Z^2 = atoms to complete a benzthiazole or naphthathiazole nucleus) can be made in several ways, e.g. by treating a dye of formula—



(R^5 = Alk or Ar) with ammonia or by heating a dye of formula—



with an organic carboxylic anhydride.
C. O. C.

Polymeric Magenta Colour Formers. General Aniline. *USP* 2,671,021

The linear polymers obtained by condensing a dihalide of a dioic acid, e.g. phosgene or oxalyl chloride with a 1-substituted-3-aminopyrazolone, e.g. 3-amino-phenylpyrazolone, to yield a bis-3-aminopyrazolone which is then treated with an aldehyde, e.g. HCHO when developed with a primary aromatic amine, e.g. 4-diethylaminoaniline, yield brilliant magentas.
C. O. C.

Carbon Black. Godfrey L. Cabot. *USP* 2,672,402

A method of simultaneously and continuously producing carbon black and a synthesis gas for liquid fuel.
C. O. C.

Readily Dispersible Satin White. Calgon. *USP* 2,671,032

Satin white is rendered more readily dispersible in water by mixing it with a protective colloid, e.g. 1–12% in its dry wt. of casein glue, and then with 0·1–3·0% of a molecularly dehydrated phosphate, e.g. sodium phosphate glass of $Na_2O : P_2O_5$ ratio 0·9:1 to 1·7:1.
C. O. C.

Red Iron Oxides. Reckitts (Colours). *BP* 717,938

Soft iron oxide pigments free of grit and deleterious salts and of exceptional brilliancy are obtained by forming an initial seed precipitate, e.g. by mixing NaOH with $FeSO_4$, and adding a finely divided oxidising agent, e.g. air, so as to oxidise the precipitate to the ferric state in 15–20 min. Metallic iron is then immersed in the liquor which is heated to about 80°c. and an oxidising agent, e.g. air, added to convert the iron into the red iron oxide.
C. O. C.

Composite Lead Chromate Lead Silicate Pigment. Titan Co. *BP* 726,191

The pigment produced by calcining a mixture of basic lead chromate, litharge and silica has excellent corrosion inhibiting properties. There should be 2–10 mol. PbO for each mol. CrO_3 and 0·75–7·5 mol. SiO_2 for each mol. PbO.
C. O. C.

Non-chalking Titanium Dioxide. American Cyanamid Co. *USP* 2,671,031

When hydrous aluminium oxide is precipitated upon calcined TiO_2 from aqueous solution and the composite pigment heated the product has much improved resistance to chalking without deterioration of other desirable pigment properties.
C. O. C.

Titanium Dioxide Pigments. Laporte Titanium. *BP* 724,751

Pigments of improved tinting strength, opacity and capacity for being wetted by and dispersed in organic paint media are obtained by dispersing the pigments in aqueous medium with the aid of a dispersing agent, then adding a water-soluble Mg salt and a water-soluble Al salt (0·1–2·0% MgO and Al_2O, respectively on the wt. of the pigment) so as not to cause flocculation, and finally adding an alkali to precipitate the Mg and Al as water-insoluble compounds in the pigment particles.
C. O. C.

Finely Divided Metallic Oxides. Deutsche Gold-und Silber-Scheideanstalt vormals Roessler. *BP* 726,250

Production of finely divided oxides with use of large flames is much simplified and cheapened by hydrolysis of metal or metallised compounds, e.g. $SiCl_4$, in a flame produced from gases which burn to produce water, e.g. H or methane and O. The gases are mixed with the vaporised metal compound in quantity sufficient to produce enough water for complete hydrolysis of the compound.
C. O. C.

Titanium Dioxide in Enamel (V below).

Aromatic Diazo Compounds. XII—Identification of Primary Aromatic Amines by means of the Absorption Spectra of the Corresponding Azo Dyes (XIV p. 274).

Rapid Testing and Numerical Evaluation of the Covering Power of Casein and Collodion Opaque Colours (XIV p. 275).

V—PAINTS; ENAMELS; INKS

Production of Colours in Titania-opacified Frits. F. J. Williams. *Finish*, **11**, (8), 43, 73; (9) 51–52, 116–117 (1954).

Titanium Dioxide in Enamel. H. J. van Buren. *Chem. Weekblad*, **50**, 605–614, 629–638 (1954): *Chem. Abs.*, **49**, 1299 (25 Jan. 1955).
Review with 209 references.
C. O. C.

PATENTS

Stable Aqueous Emulsions of Synthetic Resins which may be Pigmented. Fred'k. H. Levey Co. *BP* 722,413

Copolymers of styrene and unsaturated dibasic acids or anhydrides, e.g. maleic anhydride, are useful emulsifying and stabilising agents for preparing aqueous dispersions of other synthetic resins. Where a pigmented dispersion is required, e.g. for coating, dyeing or printing textiles, it may be used as the pigment flushing and dispersing agent. C. O. C.

Aqueous Pigment Pastes for Use in Pigmented Oil Coating Compositions. Monsanto. *BP* 717,838

A powdered water-insoluble copolymer of styrene and a half ester of maleic acid whose ester groups are methylated secondary butyl groups (1–50 parts by wt.) is added to an aqueous slurry of a pigment (100). The copolymer is then dissolved by adding ammonia, morpholine or an alkylamine volatile at $< 100°$c. followed by adding a drying oil and agitating until the pigment and copolymer are transferred to the oil phase. The mixture is then allowed to stand until the oil phase separates from the aqueous phase and can be obtained free from water by decantation. C. O. C.

Flatted and Semi-gloss Coating Compositions. Monsanto. *BP* 723,427

Silica flatting agents are prevented from hard settling if there is $\ll 0{\cdot}05\%$ by wt. of a non-ionic surface active agent dissolved in the coating composition. C. O. C.

Dry-milled Porcelain Enamels. Ferro Corpn. *USP* 2,671,029

Addition of 0·12–2·0% of a CO_3 absorbing agent, e.g. NaOH, and 0·025–0·30% of an organochlorosilane to dry-milled antimony and zirconia frits prevents formation of black specks in the finished coating even when the enamel has been kept for long periods before application. C. O. C.

Solutions of Elastomers derived from Diisocyanate-modified Polyesters—Filaments, Coating Compositions, etc. (III p. 256).
Stabilised Cellulose Ester Compositions (XI p. 272).
Determination of the Molecular Weight of Polyacrylonitrile (XIV p. 275).

VI— FIBRES; YARNS; FABRICS

Damage to Textiles from Heat Radiation. H. Berthold. *Melliand Textilber.*, **36**, 8 (Jan. 1955).

Radiation from atomic bomb explosions shows close parallels to infrared rays in absorption or reflection according to hue of the textile. Further data are given on the behaviour of other materials. S. R. C.

Transparency of Fabrics made from Different Types of Fibre. N. Duveau. *Bull. Inst. Text. France*, (50), 39–47 (Dec. 1954).

The transparency of fabrics made from natural, regenerated, and synthetic fibres has been studied—(*a*) spectrophotometrically, in the range 320–1000 mμ.; and (*b*) photographically, in ultraviolet, visible, and infrared radiation. In method (*b*) both irradiated and dark sides of the fabric were photographed. The results obtained by the two methods were in good agreement. For all the fabrics tested, the transparency increases as λ increases. J. C. F.

Concept of Swelling Anisotropy. II— Swelling Anisotropy of Different Textile Fibres. J. Pinte and P. Rochas. *Bull. Inst. Text. France*, (50), 7–15 (Dec. 1954).

The swelling anisotropies of samples of viscose rayon, cuprammonium rayon, acetate rayon, and natural silk have been measured as described previously (J.S.D.C., **70**, 587 (1954)). The coefficient of swelling anisotropy provides a measure of the macromolecular orientation of the fibre. High degrees of orientation are found for silk and cuprammonium rayon. The latter is more highly oriented than a sample of viscose rayon overstretched in spinning. Above a certain water content, dependent on the particular fibre, axial swelling reaches a constant value, and further swelling is exclusively radial. From the relative amount of radial swelling which takes place after axial swelling has ceased, conclusions are drawn concerning the state of organisation of the various fibres, i.e. the number and the stability of interchain cross-links. Acetate rayon is found to be highly organised. The organisation of cuprammonium rayon is low, which explains its accessibility to direct dyes. J. C. F.

Working Atmospheres containing Hydrogen Sulphide and Carbon Disulphide (particularly in Viscose Spinning). A. Wilke. *Faserforsch. und Textiltech.*, **5**, 513–522 (Dec. 1954).

On the basis of works experience and a critical appraisal of recommendations from many sources it is considered that for the working atmosphere of a viscose spinning plant the toleration limits are 25 mg./cu.m. for H_2S and 100 mg./cu.m. for CS_2, working limits of 10 and 70 mg. per cu.m. respectively being recommended. The ventilation requirements for the spinning machines are discussed. A. E. S.

X-Ray Diagram of Diethylacetamidocellulose Xanthate. J. Schurz. *Mh. Chem.*, **85**, 1172–1174 (Oct. 1954).

Films, stretched approx. 50% at 100–150°c. and cooled under tension, give characteristic diagrams when the number of xanthate groups per 100 glucose residues is 90–100. H. E. N.

Cold Drawing of Isotropic Cellulose Fibres. H. Klare and A. Gröbe. *Faserforsch. und Textiltech.*, **5**, 553–554 (Dec. 1954).

Isotropic cellulose fibres prepared by Hermans' method (*Kolloid-Z.*, **81**, 300 (1937)) and allowed to dry at 20°c. and 65% R.H. give the load–extension and necking behaviour that is characteristic of the cold drawing of e.g. nylon. A. E. S.

Heterogeneous Structure of Rayon. IV— Effect of Stretching— (1) and (2). S. Okajima and S. Hayama. *Bull. Chem. Soc. Japan*, **27**, 544–548, 552–553 (Nov. 1954).

(1) Properties of successive layers of a viscose rayon prepared by a two-bath process are examined. An outermost dense skin is suggested, with a less dense transition layer and core. Orientation in the skin is greater than in the core and varies directly with degree of stretching. A possible mechanism of the spinning process is suggested in terms of regeneration of cellulose from zinc xanthate at the surface, dehydration of viscose, drafting, and stretching.

(2) The structure and the orientation of chains in the outer skin and the core of a viscose rayon spun by a one-bath process are examined. W. R. M.

Old and Modern Views on the Steeping of Hemp. M. Formisano. *Ann. Fac. agrar. Univ. Napoli Portici*, **20**, 58–89 (1951–53): *Chem. Abs.*, **49**, 1333 (25 Jan. 1955).

Review with 225 references to microbiological and chemical investigations. C. O. C.

Amino Acid Composition of Keratins. I— Amino Acid Analysis of Merino 64s-quality Virgin Wool. D. H. Simmonds. *Australian J. Biol. Sci.*, **7**, 98–110 (Feb. 1954).

The ion-exchange chromatographic technique of Moore and Stein (1951) has been used to estimate the amino acids in an acid hydrolysate of merino 64s-quality virgin wool. A complete analysis of one sample of this wool is given together with figures for the C, H, N, S, and ash contents. With the exception of the glycine analyses the results confirm the figures previously acceptable as reliable for wool. P. G. M.

Amino-acid Composition of Wool. M. C. Corfield and A. Robson. *Biochem. J.*, **59**, 62–68 (Jan. 1955).

The amino acid composition of a sample of Australian Merino 64s-quality wool has been determined. The results obtained are compared with recent amino-acid analyses of wool. P. G. M.

Chemical Heterogeneity and Cortical Segmentation in Wool. R. D. B. Fraser, H. Lindley, and G. E. Rogers. *Biochim. Biophys. Acta*, **13**, 295–297 (Feb. 1954).

Further evidence for the bilateral structure of wool is given. Wool was treated with cetylsulphonic acid and then

cross-sections were stained with Toluidine Blue. One-half of the fibre was found to be stained, and termed the S segment (basophilic); the other half was unstained, and termed the H segment (acidophilic). It is suggested that the S segment may contain more glutamic and aspartic acids, and the H segment may be more highly cross-linked by disulphide bonds. When cross-sections of oxidised wool were stained with Methylene Blue, the H segment was seen to be stained and the S segment unstained. It is suggested that this may be due to the H segment containing a higher proportion of cystine. P. G. M.

Acid Hydrolysis of Wool. M. van Overbèke, G. Mazingue, and P. Desprets. *Bull. Inst. Text. France*, (50), 18–26 (Dec. 1954).

Physical tests (tensile strength, elongation at break) on fibres from samples of wool boiled in sulphuric acid solutions (solution : wool ratio 40 : 1, 8% sulphuric acid on the weight of wool) for times varying between 1 and 4 hr. showed that the mechanical properties of the wool were considerably modified when the time of boiling exceeded 3 hr. When treatment was carried out at temperatures in the range 100–125°c. (solution : wool ratio and acid concentration as before, time of treatment 30 min.), both physical tests and chemical tests (alkali solubility, acid solubility) showed that only slight degradation occurred in the range 100–115°c. Above 115°c. the amount of degradation showed a marked increase. 115°c. (240°F.) is therefore an approximate upper limit for high-temperature dyeing treatments. None of the treated samples showed a decrease in cystine content. J. C. F.

Alkaline Hydrolysis of Wool. M. van Overbèke, G. Mazingue, and P. Desprets. *Bull. Inst. Text. France*, (50), 27–37 (Dec. 1954).

Samples of wool were treated for 1 hr. at 60°c. in solutions containing 2–15 g. Na_2CO_3 per litre (solution : wool ratio 40 : 1). The following tests were made on the treated samples— alkali solubility, acid solubility, solubility in phenol–thioglycollic acid, and supercontraction in sodium bisulphite solution. The results show that only the last two tests are suitable for assessing alkali damage. The phenol solubility test has been used to study the effects of (*a*) temperature, (*b*) duration of treatment, and (*c*) concn. of Na_2CO_3 on the alkaline degradation of wool. Below 50°c. the amount of degradation is small, but above this temp. degradation rapidly increases. The effect of time of treatment is not very marked, little change taking place up to 30 min. at 50°c. with 5 g. Na_2CO_3 per litre. At temp. above 50°c. degradation increases rapidly as the concn. of Na_2CO_3 in the bath increases. J. C. F.

Structure of Silk Fibroin. R. E. Marsh, R. B. Corey, and L. Pauling. *Biochim. Biophys. Acta*, **16**, 1–34 (Jan. 1955).

An investigation based on new X-ray diffraction data, including quantitative spectrometric measurements of X-ray intensities, has led to the derivation of the fundamental structural features of silk fibroin. The structure consists of extended polypeptide chains bonded together by lateral N–H···O hydrogen bonds to form antiparallel-chain pleated sheets. The sequence –G–X–G–X–G–X–, in which G represents glycyl and X alanyl or seryl residues, predominates throughout the structure, so that adjacent sheets pack together at distances of about 3·5 and 5·7 A. Longer inter-sheet distances are explained by the presence in the structure of the larger amino-acid residues, such as tyrosine. P. G. M.

Particle Size of Renatured Silk Gel. O. Kratky, G. Porod, and A. Sekora. *Mh. Chem.*, **85**, 1176–1182 (Oct. 1954).

The diffuse small-angle scattering of X-rays is in accord with rod-like particles with a circular (diameter 80 A.) or rectangular (diagonal 107 A.) cross-section, consideration of the absolute intensity leading to corresponding values of 70 and 94 A. H. E. N.

Hydroxyethylnylon. H. C. Haas, S. G. Cohen, A. C. Oglesby, and E. R. Karlin. *J. Polymer Sci.*, **15**, 427–446 (Feb. 1955).

New derivatives of nylon are prepared by treatment with ethylene oxide. A hydroxyethyl-6,6-nylon containing 50% combined ethylene oxide is flexible and useful over a wide temperature range, having a m.p. of 221°c. and an apparent second-order transition point below —40°c. Synthetic methods, analyses, infrared spectra, viscosity behaviour, solubilities, melting points, second-order transition temperatures, and moisture permeabilities of these derivatives are reported. W. R. M.

Regain of Perlon. O. De Rix. *Textil-Praxis*, **10**, 165 (Feb. 1955).

A figure of 4% is given for the regain of Perlon compared with 2% for Orlon and 1·5% for Terylene. B. K.

Cyclic Oligomers of ε-Caprolactam. I. Rothe and M. Rothe. *Chem. Ber.*, **88**, 284–289 (Feb. 1955).

The mixture obtained in the industrial extraction of polymerised caprolactam is shown to consist of caprolactam (15%), dimer of m.p. 342°c. (36%, m.p. when pure 348°c.), trimer of m.p. 215°c. (25%, m.p. when pure 244–245°c.), and tetramer of m.p. 228°c. (13%, m.p. when pure 240°c.). The oligomers are separated by sublimation and distillation in high vacuum and repeated crystallisation from water. Mol. wt. determinations are carried out in the lactam of hexahydro-*p*-aminobenzoic acid or in acetanilide, in preference to phenol. The *N*-acetyl deriv. of the dimer has m.p. 162°c., that of the tetramer m.p. 143°c. H. E. N.

Diffusion of Water Vapour in Polymers. F. A. Long and L. J. Thompson. *J. Polymer Sci.*, **15**, 413–426 (Feb. 1955).

Diffusion of water in polyvinyl acetate obeys Fick's law at temp. of 30–50°c. and at 22°c. The diffusion coefficient is found to be independent of the concentration of water in the polyvinyl acetate. Similar behaviour is found for diffusion of water at 40°c. into cellulose acetate, cellulose nitrate, and 6,10-nylon. With polyvinyl alcohol diffusion of water is anomalous, and resembles the non-Fickian diffusion of organic vapours in cellulose acetate and cellulose nitrate. W. R. M.

Physical Structure of Asbestos. G. J. Young and F. H. Healey. *J. Physical Chem.*, **58**, 881–884 (1954): *Chem. Abs.*, **49**, 1241 (25 Jan. 1955).

In chrysotile asbestos the individual fibres are mostly bound in fibre bundles and a large portion of their external surface is not available to adsorbed molecules. It has an internal capillary structure, the capillaries appearing to be within the individual fibres and may possibly be indentified with the hollow tubular structure shown in some electron micrographs. The capillaries are blocked in some manner with strongly sorbed water plugs (these plugs are permeable to water vapour and NH_3, but not to less polar gases). The plugs are removed by activation at 425°c. at 10^{-5} mm.; they may be reformed in samples activated at < 425°c. by saturation with water vapour. C. O. C.

Surface Properties of Chrysotile Asbestos. F. H. Healey and G. J. Young. *J. Physical Chem.*, **58**, 885–886 (1954): *Chem. Abs.*, **49**, 1242 (25 Jan. 1955).

Thermodynamic functions for adsorption of gases on unactivated and activated chrysotile asbestos indicate that the unactivated surface is homogeneous to water vapour adsorption but is heterogeneous to argon adsorption. Distribution curves for the adsorption-site energies of the activated and unactivated surfaces were calculated from the isoteric heats for A adsorption. They indicate that the surface of the capillary structure made available in activation is of lower energy in regard to non-polar gas adsorption than the external surface of the fibres. Heats of immersion for the asbestos–water system support the conclusion that the unactivated asbestos surface is homogeneous for polar gas adsorption. C. O. C.

PATENTS

Reducing the Viscosity of Solutions of Polyacrylonitrile in Organic Solvents. Chemstrand Corpn. *USP* 2,671,066

Addition of a little dimethylamine hydrochloride, hydroxylamine hydrochloride, phenylhydroazine hydrochloride or aniline sulphate to an organic solvent used for dissolving polymers containing $< 80\%$ of acrylonitrile results in solutions of appreciably lower viscosity than those formed in absence of the amine salt. C. O. C.

Chlorine-containing Vinyl Polymeric Compositions. Société des Usines Chimiques Rhône-Poulenc. *BP* 725,590

Removal of the liquid from a dispersion or solution containing a chlorine-containing vinylpolymer and a homopolymer of a compound of formula—

$$CH_2{:}CH{\cdot}O{\cdot}CH_2CH_2NR^1R^2$$

(R^1 and R^2 = H or same or different, satd. or unsat. aliphatic hydrocarbon or together with the N atom form a heterocyclic ring), e.g. vinyl ether of *N*-hydroxyethylpyrrolidine, leaves a product which has good affinity for acid and metalliferous dyes. The dyed product has excellent fastness to washing. C. O. C.

Polyacrylonitrile—Polyaminoethylvinyl Ether Mixtures having Affinity for Acid Dyes. Société des Usines Chimiques Rhône-Poulenc. *BP* 725,569

The product obtained by evaporating a solution containing a polymer-containing nitrile groups and a homopolymer of a compound of formula—

$$CH_2{:}CH{\cdot}O{\cdot}CH_2{\cdot}CH_2{\cdot}NR^1R^2$$

(R^1 and R^2 = H or hydrocarbon or together make an alkane chain which with the N atom form a heterocyclic nucleus), e.g. the vinyl ether of dimethylaminoethanol, is readily dyed with acid dyes. C. O. C.

Dye-receptive Acrylonitrile Polymers. Chemstrand Corpn. *USP* 2,671,072

Copolymers of acrylonitrile and α-chloroacrylic acid derivatives or blends of polyacrylonitrile with such derivatives when treated with agents which convert the chlorine atom into an ionic chlorine radical, e.g. ammonia, amines, thiourea or 2-mercaptobenzothiazole, have much increased affinity for acid dyes. C. O. C.

Highly Adsorbent and Water-absorbent Artificial Fibres. F. D. Timmermans. *BP* 723,959

Highly water-absorbent artificial fibres, particularly suitable for making bandages, are made by extruding spinning solutions to which has previously been added finely ground silica gel and a suitable alkali-stable dispersing agent. J. W. B.

Felted Glass Fibre Material. Hawley Products. *BP* 723,955

Glass yarns are treated with a bonding agent, preferably a composition consisting essentially of polyvinyl acetate and a plasticiser, e.g. dibutyl phthalate, the coating then being hardened by heat-treatment. The yarns are then chopped into lengths, e.g. 0·5–6·0 in., and, along with other fibrous material, e.g. wool, cellulosic, lignocellulosic or nylon, are made into an aqueous slurry. The glass filaments do not disperse and the pieces of yarn thus remain whole. The quantity of glass should be 5–35% of the whole, and a small proportion of water-dispersible glass fibres may be included. Useful accreted moulded fibrous products can be formed from these slurries. J. W. B.

Bonding Fibres Together. Chavannes Industrial Synthetics. *USP* 2,671,496

A powdered thermoplastic resin is blown into corded laps which are then arranged in layers, each lap being at right angles to the adjacent laps. The assembly is then passed through pressure rollers, heated and then passed through a cooling zone with the upper end lower surfaces of the assembly held parallel. C. O. C.

Tint Removal from Staple Fibres (VIII below).

Determination of the Molecular Weight of Polyacrylonitrile (XIV p. 275).

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Foaminess of Aqueous Solutions. L. Topper and E. Baer. *J. Colloid Sci.*, **9**, 495–497 (Dec. 1954).

Studies of the foaminess of aqueous solutions of sodium acetate, sodium propionate, and sodium *n*-butyrate and of a 0·025% suspension of 30-mμ. silica particles show that both maximum foam height and maximum collapse time are associated with the same concentration of solute. Addition of silica does not change this concentration appreciably, but extends the concentration range of good foam height and stability. W. R. M.

PATENTS

Bleaching with Sodium Chlorite. Val Mehler Segeltuchweberei. *BP* 723,566

The material is impregnated continuously and preferably in open width with aqueous alkali metal chlorite, then treated for a short time, usually 1–2 min., with acid vapour. This causes formation of chlorine dioxide *in situ* in the exact amount required, avoids any chance of injury to the workmen and avoids the necessity of expensive suction equipment. C. O. C.

Bleaching Textiles. DuP. *USP* 2,670,266

The materials are impregnated with a finishing agent and Na chlorite or an organic peracid or an alkali metal salt of an organic peracid and dried. This enables bleaching and finishing to be carried out in a single operation. It is especially suitable for treating viscose filaments during their manufacture. C. O. C.

Bleaching Polyamide Fibres. N.V. Onderzoekingsinstituut Research. *BP* 725,953

Bleaching is carried out with no loss of strength by first treating with aqueous alkali metal or alkaline earth metal hypochlorite and then with a reducing agent at $> 80°$C. C. O. C.

VIII—DYEING

Influence of the Physical State of Dyes upon their Light Fastness. G. Baxter, C. H. Giles, M. N. McKee, and N. Macaulay. J.S.D.C., **71**, 218–235 (May 1955).

From a study of the quantitative relationship between light fastness and dye concentration conclusions are drawn regarding the physical state of dyes in air-dry substrates, on the basis of a simple theoretical model representing the nature of particle growth with increase in concentration.

Only one type of dye–substrate system, viz. a water-insoluble *merocyanine* dye in solid solution in collodion, has been found to obey the normal photochemical laws for ideal systems. All other systems, whether involving water-insoluble or water-soluble dyes, behave non-ideally: i.e. the dye appears to exist in the form of aggregates or crystals (usually submicroscopic) and to obey a fading law in which the rate-controlling factor is the extent of the dye–air interface. Treatments which cause microscopically visible growth of such particles, e.g. soaping of azoic-dyed cellulose, raise the light fastness as anticipated.

Also in accordance with prediction, the light fastness even of water-soluble dyes is raised by increasing the porosity of the substrate; this is believed to result from the formation of larger aggregates of dye in the dry substrate. Thus the light fastness of direct cotton dyes is higher on highly porous ("gel") Cellophane film than on normal film, and on normal viscose rayon fibre than on highly oriented viscose rayon fibre; and acid wool dyes have higher fastness on the sulphuric-acid anodic film on aluminium than on the less porous chromic-acid film. A number of other examples of changes in light fastness, believed to be due to similar physical factors, are discussed.

The following two observations are reported—(*a*) undried "gel" Cellophane film slowly reverts to the normal form on keeping, even when wet; and (*b*) there is a linear relationship between the logarithm of percentage reflectance and the logarithm of total dye content for direct dyes on various viscose rayon fabrics. AUTHORS

Tint Removal from Staple Fibres. AATCC, Philadelphia Section. *Amer. Dyestuff Rep.*, **44**, P 41–P 53 (17 Jan.1 955).

Tint removal is becoming complicated by the tendency of new processing operations to fix many dyes and pigments in the fibres. To study these effects continuous-filament Saran and staple yarns of cotton, acetate and viscose rayons, wool, silk, Vicara, nylon, Dacron, Acrilan, Orlon 42, and dynel were placed in successive groups of ends in a warp, the weft being Orlon 81. Tapes of each

fibre were woven separately. Four dyes and three pigments were selected, and were applied by padding, no auxiliaries being used. Samples of the fabric were subjected to heat setting, crimp setting, can-drying and ageing, the fact that certain fibres would thereby be severely damaged being ignored, though details of such damage are given. Scouring was carried out under different conditions of pH and temp., and with different detergents. Results are tabulated in terms of tinctorial strength of the tints. On the unprocessed control all four acid dyes were removed by one or another method, some difficulty being found with Amido Naphthol Red 6B (*C.I.* 57) on wool, cotton, and nylon and with Wool Green S (*C.I.* 737) on wool and nylon. All the pigments were more difficult to remove, the greatest trouble being experienced with Algol Yellow GC. Heat setting made removal of Tartrazine (*C.I.* 640), Amido Naphthol Red 6B, and Wool Green S from nylon more difficult; both Acrilan and Orlon 42 retained Wool Green S, and Amido Naphthol Red 6B was more difficult to remove from Acrilan and to a lesser extent from Orlon 42. Heat setting had no significant effect on Indigotin 1A (*C.I.* 1180). Crimp setting led to greater difficulties, notably with Amido Naphthol Red 6B and Wool Green S from wool, Vicara, nylon, Acrilan, and Orlon 42, and Tartrazine from nylon. Even Indigotin 1A became difficult and was essentially non-removable from, Acrilan by any method. Can-drying had little effect. Ageing inhibited removal of Wool Green S from wool, Vicara, nylon, Acrilan, and dynel, and of Amido Naphthol Red 6B from nylon and (slightly) Vicara. J. W. B.

Adsorption at Organic Surfaces. I— Adsorption of Organic Compounds by Polyamide and Protein Fibres from Aqueous and Non-aqueous Solutions. H. R. Chipalkatti, C. H. Giles, and (the late) D. G. M. Vallance. *J.C.S.*, 4375–4390 (Dec. 1954).

The adsorption is recorded of a range of aliphatic and aromatic non-ionic cpd., on nylon and wool, from a variety of solvents. In aq. soln., all the water-soluble cpd. used have unrestricted access to the non-cryst. regions of the fibres by virtue of the swelling action of the solvent, and adsorption is probably due to hydrogen bonds. In non-aq. soln., the fibres are unswollen and the molecules of solutes which are non-solvents for the fibre are adsorbed only in certain circumstances, which are— (*a*) that they are within certain limiting dimensions, viz. ca. those of the *n*-butanol mol. and the anthracene mol. in the aliphatic and aromatic series respectively; and either (*b*) that they contain a free OH group or an extended conjugate system; or (*c*) that the solvent in which they are dissolved is itself unable to penetrate the fibre. The evidence suggests that from non-aq. soln. OH cpd. are adsorbed by the formation of O · · · H · · · O hydrogen bonds, probably with the enolic forms of amide or peptide groups of the fibre, whereas with non-OH cpd. physical adsorption occurs. H. H. H.

Adsorption at Inorganic Surfaces. I— Mechanism of Adsorption of Organic Compounds by the Anodic Film on Aluminium. C. H. Giles, H. V. Mehta, C. E. Stewart, and R. V. R. Subramanian. *J.C.S.*, 4360–4374 (Dec. 1954).

The chromic-acid anodised film on Al is positively charged in H_2O, and anionic but not cationic cpd. are adsorbed; non-ionic compounds of suitable structure may also be adsorbed from aq. or non-aq. soln. The film consists of substantially pure γ-alumina, and the following adsorption mechanisms operate in aq. soln.— (*a*) salt formation between the metal of the film and certain sulphonated cpd., (*b*) ion-exchange at the surface with some sulphate esters, (*c*) hydrogen-bond formation between the oxygen of the film and the hydrogen atom of phenolic groups, (*d*) chelation between the metal of the film and suitable pairs of *ortho* substituents in the solute; and in non-aq. soln.— (*e*) hydrogen-bond formation with phenols and amines, and (*f*) a "bridge-bonding" mechanism by which some donor solutes can be adsorbed. Donor cpd. are adsorbed only by (*f*). The protective action of the solvent can also prevent adsorption of many solvents, e.g. some sulphonates from H_2O, and amines from C_6H_6 or H_2O. H. H. H.

Factors which influence Fastness to Crocking [Rubbing]. AATCC, New York Section. *Amer. Dyestuff Rep.*, 44, P 34–P 40 (17 Jan. 1955).

Problems due to lack of rubbing fastness and methods of measuring it are surveyed, and a study is made using four commercially dyed cotton fabrics (two azoic and two sulphur dyeings) and a number of laboratory specimens (vat dyes applied by three methods, azoics by padding and jig developing, and printing with vats, azoic, and pigments). Some aftertreatments are also applied, including an extra scour, a melamine–formaldehyde resin, a synthetic rubber latex, and a silicone resin. Rubbing fastness has been assessed at five different laboratories, the results being presented graphically. General observations that lower ratings are obtained with wet rubbing than with dry rubbing are confirmed. The fastness of vat dyeings to wet rubbing is improved by superior preparation (mercerisation, boiling off, and bleaching) of the substrate, which, however, has little effect on the dry fastness of vat dyeings and on the wet and dry fastness of prints. Vat dyeings are in general better than vat prints, which in turn rate higher than azoics, and are best when applied by the Williams method. The pad–steam procedure is inferior with regard to both wet and dry rubbing. Aftertreatments on black sulphur-dyed and maroon azoic-dyed fabrics lead to no improvement, the silicone resin actually having an adverse effect on the black. J. W. B.

Dyeing of Tops with Preservation of Spinning Properties. K. Wojatschek. *Melliand Textilber.*, 36, 68–71 (Jan. 1955).

The effects of pH, time of dyeing, and fibre-protective agents are examined. Acid, metal-complex, metachrome, and afterchrome dyes are discussed. S. R. C.

Dyeing Characteristics of Different Wool Grades. W. F. Brommelsiek, W. von Bergen, and H. E. Millson. *Amer. Dyestuff Rep.*, 44, P 73–P 86 (31 Jan. 1955).

Dyeing properties of 4 wools— an Australian Fine, a New Zealand Medium, a Domestic Fine, and a Domestic Medium— are studied. Dyeings are made on piece, top, and individual staple, and include acid, acid metallised, and chrome dyes. There is considerable variation in shade, analysis revealing that shade differences are directly related to fibre fineness. One wool-grade difference is equal to a 5% difference in depth of colour. Spectrophotometric analysis of dye liquor shows no difference in total exhaustion but slight differences in rates of exhaustion. Wool dyeings which are level during the low-temp. period are not necessarily level at the end of the boil, whereas early poor penetration may be followed by an ultimately uniform result. It is also shown that the transmission of light through a uniform cross-section of fine or medium wools shows the reverse contrast to the reflectance from pigment pastes of the same fibres pulverised. J. W. B.

Dyeing Reactions. VI— Absorption of Palatine Fast Dyes by Modified Protein Fibres. H. G. Fröhlich. *Z. ges. Textilind.*, 55, 1267–1273 (1953): *Chem. Abs.*, 49, 1332 (25 Jan. 1955).

Combination between the dye and the fibre is dependent on the presence in the fibre of free carboxyl and amino groups, especially the primary lysine amino group. Blocking or destruction of these groups by acetylation or deamination decreases the amount of dye absorbed. Absorption of the dye increased with decrease of pH, maximum absorption in wool, silk and Ardil being at pH 1–2 and with zein fibre at pH 3–4. Wool, silk and Ardil showed a second maximum dye absorption at pH 6–7, this second maximum being absent from zein fibre which contains no lysine amine groups and from acetylated or deaminated fibres. Absorption of dye by polyamide fibres and absorption of Cr from chrome-alum solution by untreated protein fibres confirmed these results. C. O. C.

Dyeability of Modern Synthetic Fibres. J. G. Kern. *Amer. Dyestuff Rep.*, 44, P 22–P 28 (3 Jan. 1955).

The chemical structures of wool and modern synthetic fibres are described and compared, and a survey is presented of dyeing methods. J. W. B.

Effect of Chlorine on Dyed Nylon. A. N. Davidson and R. Preston. J.S.D.C., **71**, 235–243 (May 1955).

After dry chlorination of wool, the chlorine retained by the fibres is present as hydrochloric acid (i.e. is extractable by alkaline solutions); whilst with nylon it is shown that the chlorine is more strongly combined, so that it is not completely extracted by sodium carbonate solution but can be removed by sodium bisulphite solution. Generally, absorption of chlorine by nylon is attended by an increase in fluidity of the nylon in *m*-cresol and a corresponding decrease in tensile strength; the degree of absorption of chlorine appears to vary with the type of dye used. The presence of wool has a "buffering" action on the uptake of chlorine by nylon and hence tends to protect the nylon from excessive damage. Of the groups of dyes examined (chrome, level-dyeing and neutral-dyeing acid dyes, together with a few soluble acetate rayon dyes and direct cotton dyes), the chrome dyes were satisfactory in their fastness to chlorination. The lighter shades of neutral-dyeing acid dyes were useless and the other dyeings occupied intermediate positions. All these dyes, with a few exceptions, have a slightly higher fastness to wet chlorination. The above observations are illustrated in detail by tabulated and graphed data, together with colorimetric data for a representative selection of the dyes before and after dry chlorination. AUTHORS

Use of Diammonium Phosphate in Dyeing Dacron and Dacron–Wool Blends. G. W. Spicer. *Amer. Dyestuff Rep.*, **44**, P 87–P 88 (31 Jan. 1955).

The Na salt of *o*-phenylphenol is effective as a carrier in dyeing Dacron polyester fibre, particularly in blends with wool. It is improved by employing diammonium phosphate for converting the water-soluble form to the insoluble *o*-phenylphenol: there are no extreme pH changes during the dyeing cycle, the buffering effect conditions the wool for dyeing, the dispersing effect of diammonium phosphate eliminates the need for other dispersing agents, and colour values are increased. J. W. B.

Dyeing Dacron Fabrics by the Carrier Method. Z. L. Collins, L. G. Darby, G. A. Harris, and J. H. Howell. *Amer. Dyestuff Rep.*, **44**, P 29–P 30 (3 Jan. 1955).

A laboratory study is made of seven carriers for the application of disperse (acetate-rayon) dyes to Dacron polyester fibre. In general all the carriers lead to an improvement, though what is best for one dye is not necessarily best for another. The most satisfactory carriers are 15% β-naphthol, 20% benzoic acid, 15% benzyl alcohol, or a combination of 5% benzoic acid and 10% β-naphthol (all calculated on wt. of fibre). β-Naphthol is somewhat difficult to remove from the fabrics after dyeing, but its toxicity may not matter if the reagent is used in a closed, well ventilated machine. Acetophenone is found to be effective in conjunction with Dowicide A and β-naphthol in increasing the penetration. A useful modification is brought about by prepadding with a concentration of dye to give 2% pick-up, and then entering into a hot bath containing 2% disperse dye, 15% Dowicide A, and 2% acetophenone, plus acetic acid to a pH of 5. The liquor is boiled for 1 hr., care being taken to keep it on the acid side, and the dyeing is after-scoured in Hydros and NaOH. J. W. B.

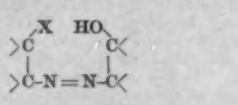
Surface Treatment and Finishing of Light Metals. V— Chemical Conversion Coatings and their Dyeing. S. Wernick and R. Pinner. *Metal Finishing*, **52**, (10), 68–73; (11), 83–87 (1954): *Chem. Abs.*, **49**, 818 (25 Jan. 1955).

Oxide conversion coatings are much thinner than the oxide film produced by anodising. Their main use is as an undercoating and a base for organic finishes. The production of these coatings, the effects on colour of the agents used to produce them, and their dyeing are discussed. 26 references. C. O. C.

PATENTS

Improving the Wet Fastness of Dyeings on Prints made with Direct Dyes. Ciba. *BP* 722,321

The wet fastness of direct dyes contaning sulphonic acid or carboxylic acid groups can be improved by after-treatment with an aqueous solution of the condensate of an aldehyde with the product obtained by heating at $> 160°$c. dicyandiamide with a salt of an aliphatic or *cyclo*aliphatic amine containing > 1 primary or secondary amine group. The treatment can be combined with an aftertreatment with a water-soluble copper compound, this being especially advantageous where dyes containing the



(X = OH or COOH the pair of C atoms to which it is bound forming part of a cyclic diazo component; the pair of C atoms to which the OH group is bound forms part of any desired coupling component.) C. O. C.

Sulphaminic or Aromatic Sulphinic Acid for use in Dyeing with Acid Dyes. FH. *BP* 723,928

Much brighter dyeings of good fastness properties are produced by using sulphaminic acid or an aromatic sulphinic acid, $Ar{\cdot}SO_2H$, or a salt thereof, instead of the usual sulphuric or formic acid. C. O. C.

Dyeing Cellulose Esters with Vat Dyes. DuP. *USP* 2,670,264

The cellulose ester is treated with a polymeric amino-alcohol ester of acrylic acid or an α-substituted acrylic acid, e.g. cellulose acetate is impregnated with 7% of poly-dimethylaminoethyl methacrylate in a mutual solvent. The modified material is then treated with a salt of an acid ester of a leuco vat dye, rinsed, oxidised, neutralised, scoured and dried. C. O. C.

Dyeing Polyamide Fibres of Differing Denier. N.V. Onderzoekingsinstituut Research. *BP* 725,390

Fabrics containing polyamide fibres of different titres dye solid with disperse dyes if the bath is maintained at pH 3·0–5·5. C. O. C.

Pigmented Resin-Drying Oil Compositions for Application to Glass Fibre Fabrics. Sun Chemical Corpn. *USP* 2,671,738

A mixture of a drying oil, an oil-soluble rosin-phenol-formaldehyde condensate, an insoluble pigment opd., a drier which is also insoluble in the drying oil and a diluent is used to impregnate the fabric. After drying the impregnated fabric shows a pattern of variegated depths of colour varying from almost complete absence of pigment to the colour of the solid pigment. C. O. C.

Pastes for Coating, Printing, Finishing and Imparting Water-repellency (III p. 256).
Polymerised Ethyleneimine as a Gas Fume Fading Inhibitor (III p. 256).
Progress in Dye Manufacture and in Dyeing, Printing, and Finishing in the U.S.S.R. (IV p. 257).
Acceptability and Perceptibility of Fading (XIV p. 275).

IX— PRINTING

Printing of Azoic Dyes and Appropriate Thickening Agents. J. Hausermann. *Teintex*, **20**, 13–19 (Jan. 1955).

Rapidogen types are considered. The non-reducing modified carob flour is favoured with and without starch. The effect of copper in inhibiting coupling and precipitating thickenings can be avoided by adding appropriate products to the pastes. S. R. C.

PATENTS

Preventing Hardening of Resinous Printing Pastes on Printing Blankets. Dewey & Almy Chemical Co. *BP* 722,674

An endless printing blanket has a working surface of textile material partly embedded in an elastically deformable cushioning layer. After the printing operation it is washed, dried, a solution of rubber extender being continuously applied to the blanket after it leaves and before it re-enters the printing position. Application of the rubber extender prevents the hardening of any resinous printing paste adhering to the blanket no matter how long it is heated at curing temperature. C. O. C.

Transfers. Omega Plastics. *BP* 723,776

A film backing layer carries a design printed wholly or in part with a fluorescent ink, the areas in fluorescent area being underprinted with a highly reflective layer of either a light coloured or white pigment or a pigment of the same hue as the fluorescent ink. The whole is secured by a removable adhesive layer to a temporary support. C. O. C.

Colour Photography. ICI. *BP* 723,171

A simple method of forming a masking image from residual colourless colour former in the cyan layer, e.g. phenols and naphthols having no substituent in the 4-position to the OH group, without undesirably affecting the primary or masking images in the other layers. After colour development the image is treated at any stage with HCHO in presence of a primary aromatic amine and the resulting leuco derivative of the masking dye is oxidised. C. O. C.

Sensitive Elements for Securing Colour Correction in Colour Photography. Kodak. *BP* 726,377

Imbibition Printing of Cinematograph Film. Technicolor Motion Picture Corpn. *BP* 726,218

Means for overcoming loss of film due to poor transfer adjacent hills in the matrix relief, to increase the rate of dye transfer and to improve definition of the imbibition prints by use of film having harder dye-absorptive material. C. O. C.

Diazotype Material. Kalle & Co. *BP* 722,844

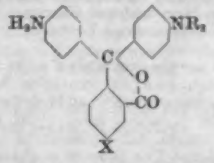
A coloured water-insoluble *o*-diazophenanthol or an *o*-quinonediazide of an aromatic sulphone which bleaches out under the influence of light is used as the light-sensitive constituent, the exposed material being developed with dilute alkali to yield a positive image from a positive original and a negative image from a negative original. C. O. C.

Azo Dye Images in Photographic Materials. General Aniline. *USP* 2,671,023

A negative or print bearing a silver halide image is treated with an alkaline solution of a colourless compound containing a radical permitting it to react with the silver halide to form a less soluble silver salt and also a group permitting it to couple with a diazonium compound to form a dye, e.g. 2-thiobarbituric acid or 5-hydroxy-*α*-naphthylthiourea. It is then thoroughly washed with water and treated with a diazonium compound free from water-solubilising groups other than the diazonium group. It is then washed to leave clear whites and a coloured image which is insoluble in the usual silver salt solvents. The process is particularly applicable with use as the diazonium chloride of the zinc chloride double salt of the diazonium chloride prepared from either 2-amino-4-chlorophenyldioxide or 1-aminoanthraquinone. C. O. C.

Eradication of Marks from Reactant Record Material. National Cash Register Co. *USP* 2,672,396

Coloured marks formed by adsorption of a colour-reactant organic compound, e.g. a compound of formula—



(R = Alk of < 5 C; X = H or dialkylamino of < 9 C), e.g. into a material sensitised with an acid-like electron-acceptor adsorbent pigment, e.g. attapulgite, are removed by applying a volatile acid, e.g. acetic acid or hydrochloric acid, preferrably dissolved in acetone, methyl ethylketone or ethylacetate. C. O. C.

Rendering Electrostatic Images Visible. Ferranti. *BP* 723,539

An electrostatic image formed by photographic exposure of a uniformly charged surface of a layer of photo-conductive insulating material coated on a conductive surface of a plate is developed in darkness by forming a powder into a cloud, passing this cloud through a metallic mesh connected to a source of fixed potential into contact with the photo-conductive layer while the latter is connected to a different source of fixed potential. C. O. C.

Magnetic Reproduction of Pictures. R. B. Atkinson. *BP* 725,142

A flowable pigment-like material, e.g. iron oxide dispersed in oil, is applied to a magnetic record of an image. C. O. C.

Pastes for Coating, Printing, Finishing and Imparting Water-repellency (III p. 256).

Progress in Dye Manufacture and in Dyeing, Printing, and Finishing in the U.S.S.R. (IV p. 257).

X—SIZING AND FINISHING

Absorption of Size by Cotton Warps. K. Ramaszéder. *Textil-Praxis*, **10**, 38–44 (Jan. 1955).

The absorption of size by warps during sizing depends on several factors— quality, twist, count, thickness, and wettability of the cotton yarn; condition and viscosity of the size; extent of dilution of the sizing liquor due to condensation of steam used for heating; depth of yarn in sizing trough; hardness of covering on squeeze rollers and their weight; rate of passage of warp through size. A fundamental investigation into the mechanical factors affecting sizing is described. B. K.

Wrinkle-resistant Finish for Cellulose Fabrics. T. F. Cooke. *Amer. Dyestuff Rep.*, **43**, P 891–P 896 (20 Dec. 1954).

It is suggested that urea– and melamine–formaldehyde resins impart resistance to the wrinkling of cotton fabrics by increasing the elasticity of the fibres, which confers increased elasticity on the yarns and thus on the fabric. A number of different resins are rated in the following order of decreased wrinkle resistance— combination of melamine–formaldehyde and styrene–ethyl acrylate co-polymer, melamine–formaldehyde, styrene–ethyl acrylate copolymer, and finally polyvinyl acetate and the lactic acid colloid of melamine–formaldehyde, both of which give poorer results than the control. A new accelerator, Aerotox Accelerator MX (American Cyanamid Co.), is found to improve the wrinkle resistance of fabrics treated with melamine–formaldehyde resin, and to reduce yellowing and loss in strength when such fabrics are chlorine-bleached. J. W. B.

Theoretical Aspects of the Flameproofing of Cellulose. H. A. Schuyten, J. W. Weaver, and J. D. Reid. *Advances in Chem. Series*, No. 9, 7–20 (1954): *Chem. Abs.*, **49**, 1322 (25 Jan. 1955).

The theoretical aspects of imparting flame-resistance to cellulose are discussed mainly on the basis of practical work by using the modern concepts of organic chemistry to explain the mechanism of the chemical reaction involved. Only one chemical mechanism, dehydration, aided by a physical mechanism, accelerated gas production, explains the behaviour of all known flame-resisting agents. Flame-resistance results from the catalytic dehydration of cellulose through the outer reaction of the flame-resisting agent via a carbonium mechanism probably identical with that advanced for the dehydration of monohydric alcohols and glycols. The flame-resisting agent may either be present at the start or else it is produced from its "precursor" at a temperature close to that of burning cellulose. It must not be volatile at 300–350°c. and must not burn readily. It must be a "Lewis acid" or capable of forming a "Lewis acid" at the temperature of burning cellulose. C. O. C.

Flameproofing of Textiles. W. Stobowsky. *Teintex*, **20**, 113–119 (Feb. 1955).

One-bath treatments in (*a*) phosphates with or without melamine resins or antimony salts, (*b*) acid solutions of titanium and antimony chloride, (*c*) inorganic salts, e.g. $CaCO_3$, dispersed in chlorinated paraffins or chlorinated rubber, (*d*) polyvinyl chloride and antimony oxide with or without chlorinated paraffin are recommended. S. R. C.

Flameproofing of Camouflage Net. W. Rudolf. *Melliand Textilber.*, **36**, 75–79 (Jan. 1955).

Specifications of the finished article, including test methods and fabric types, are discussed. A number of soluble and dispersed flameproofing agents are considered critically. S. R. C.

Durable Combined Finishes on Textiles. A. Gervie. *Teintex*, **20**, 20–31 (Jan. 1955).

The possibilities of combining fire-, water-, and rot-proofing are examined. S. R. C.

Melamine Resins in Textile Finishing. F. Liewald. *Textil-Praxis*, **10**, 79–86 (Jan. 1955).

The application of an unmodified and a partly modified hydroxymethylmelamine resin (Cassurit MKF conc. powder and MLP liquid (CFM)) for the production of permanent embossed, silk, and chintz finishes on cotton and rayon staple is described. B. K.

Urea–Formaldehyde Condensation. IX—The Presence of Dimethylene Ether Bridges in Condensates. G. Zigeuner, K. Voglar, and R. Pitter. *Mh. Chem.*, **85**, 1196–1207 (Dec. 1954).

Phenylurea reacts with formaldehyde to form the *N*-hydroxymethyl deriv. in alcoholic NaOH, but yields the dimethyl ether $(C_6H_5{\cdot}NH{\cdot}CO{\cdot}NH{\cdot}CH_2)_2O$ in aq. K_2CO_3. Both types of cpd. are converted into alkyl phenylureamethyl ethers $C_6H_5{\cdot}NH{\cdot}CO{\cdot}NH{\cdot}CH_2{\cdot}O{\cdot}Alk$ by alcohols and a little HCl, but these reagents leave methylenebisphenylurea $(C_6H_5{\cdot}NH{\cdot}CO{\cdot}NH)_2CH_2$ unaltered. H. E. N.

Action of Unsaturated Aldehydes on Wool. J. R. McPhee and M. Lipson. *Australian J. Chem.*, **7**, 387–396 (1954): *Chem. Abs.*, **49**, 1333 (25 Jan. 1955).

Wool treated with acraldehyde (I) 0·1–5·0% solutions v/v in buffers of pH 0·9–10·8 has its alkali solubility substantially lowered, optimum effect being obtained with 60 vol. 0·5% v/v I in 0·2 M-phosphate buffer (pH 8·0) at 50°C. for 6 hr. Under these conditions acraldehyde reacted to a greater extent than α-methacraldehyde (II) and crotonaldehyde (III) and decreased the alkali solubility the most. Acetylated wool is much less affected than normal wool. That there is little reaction with the imino groups of the main chain is shown by the fact that when nylon is treated with I there is only slight increase in weight. Wool treated with I has better resistance than untreated to boiling for 1 hr. in 0·1N-HCl. Treatment with I had no effect on the supercontraction caused by boiling $NaHSO_3$, but halved that caused by LiBr; it did not affect the 30% indexes of single fibres in water. Wool reduced with 60 times its weight of 0·2 M-thioglycollic acid for 18 hr. at 22°C. had increased affinity for I, II, III and acrylonitrile (IV); I and IV had the greatest action on the cysteine of the fibre to form bonds stable to acid hydrolysis. Reduced wool treated with I and II did not supercontract in boiling $NaHSO_3$; III was only partly effective and cinnamaldehyde (V), IV and acetaldehyde (VI) had no effect. Reduced wool treated with I and II was highly resistant to moth grubs. Cysteine and cystine analysis and paper chromatography showed that IV, V, and VI have little effect on normal wool. II and III have some effect but not enough to cause detectable changes in amino-acid composition. I has the greatest effect and combines with free SH and ε-NH_2 groups. Free aldehyde groups are present after the treatment. The probable reactions are—

$$-CH_2NH_2 + CH_2{:}CHCHO \rightarrow -CH_2NHCHOHCH{:}CH_2$$
$$-CH_2SH + CH_2{:}CHCHO \rightarrow -CH_2SCH_2CH_2CHO$$

If the same molecule of acraldehyde reacts with both lysine and cysteine groups, new cross links will be formed. This reaction is probably slight with normal wool but much greater with reduced wool. The main effect may be steric, as blocking of NH_2 groups adjacent to S–S bonds may reduce the lability of the latter. C. O. C.

Digestion of Wool by Clothes-moth Larvae. E. H. Mercer. *Biochim. Biophys. Acta*, **15**, 293–295 (Oct. 1954).

The first portion of the wool fibre to be attacked by clothes-moth larvae is probably the orthocortex, a hemi-cylindrical segment of the cortex which is less stabilised and more readily attacked by many reagents. The cuticle and the remaining portion of the cortex (para-cortex) are more resistant and may escape digestion entirely when the larva is feeding rapidly. There is some evidence that the digestion of wool by fungi and bacteria follows a similar course. P. G. M.

Production of Mothproofed Wool by Chemical Modification. H. Zahn and A. Räuchle. *Textil-Praxis*, **10**, 89–93 (Jan. 1955).

Good results are obtained by using epichlorohydrin, acetic anhydride, bischloromethyldimethylbenzene, *p*-bromo-ω-bromoacetophenone, quinone, and 1:5-difluoro-2:4-dinitrobenzene. Wool mothproofed by the foregoing methods has been examined for resistance to exposure to light, effect of alkalis, and dyeing properties. B. K.

PATENTS

Sized Untwisted Rayon Threads. Vereinigte Glanzstoff-Fabriken. *BP* 723,576

Flattening of the threads is removed if after passing through the squeeze rollers they pass through a groove in another roller. The cross-sectional dimensions of this groove depend upon the denier of the thread and are such that the thread or group of threads engages the sides of the groove so as to change the flat form of the thread or group of threads to a round cross-sectional form. C. O. C.

Sizing of Paper. DuP. *BP* 726,364

The lignocellulosic fibres in the beater stage of paper manufacture are subjected to the action of ethylene homopolymer, in the form of solid particles of sub-microscopic size colloidally dispersed in an aqueous medium with an alkali-metal laurate, while the dispersed homopolymer is coagulated with alum. After the dispersion has been added, the beater stock is kept at pH 5·5–6·0, and the beating is continued until the finished paper will contain 0·1–5·0% of the polymer to effect sizing of the paper. S. V. S.

Setting Tubular Fabrics of Cellulose Acetate. Adshead & Geeson. *BP* 722,461

The process of *BP* 683,075 (J.S.D.C., **69**, 105 (March 1953)) can be used for fabrics containing cellulose acetate either alone or mixed with other fibres. C. O. C.

Dimensional Stabilisation of Cellulose Fabrics. Fothergill & Harvey. *BP* 724,096

Modification of *BP* 696,282 (J.S.D.C., **69**, 390 (Oct. 1953)). Addition of an alkali metal carbonate to the aqueous alkali used enables the concentration of caustic alkali to be lowered and/or the time of treatment to be shortened. C. O. C.

Impregnating Cellulose Fibres with Sericeous Material. T. C. Whitner. *USP* 2,671,740

Cellulose fibres are impregnated with a solution of silk, e.g. with the solution prepared according to *USP* 2,646,372 (J.S.D.C., **70**, 426 (Sept. 1954)) and then treated with aqueous acid. C. O. C.

Increasing Durability and Snag-resistance of Textiles made from Non-animal Fibres. Commonwealth Scientific and Industrial Research Organisation. *BP* 726,263

The materials are treated with a solution in an organic solvent of a high mol. wt. synthetic linear polyamide substituted by alkoxy methyl or hydromethyl groups on the carbonamide N atoms and then hydrolysing the substituted polyamide, e.g. by treating with an aqueous acid or by heating. The process is particularly suitable for imparting snag-resistance to knitted fabrics. C. O. C.

Reducing the Affinity of Protein Fibres for Acid or Neutral Wool Dyes. S. *BP* 723,690

Protein fibres are given excellent resistance to dyes which normally dye them from acid or neutral baths by impregnating them with an acid solution of sulphamic acid or a derivative of it containing the $-NHSO_3H$ group and which splits off sulphamic acid on heating, drying at < 100°C. and then heating the impregnated fibres at 120–175°C. until reaction takes place between them and the sulphamic acid. C. O. C.

Reducing the Tendency of Wool to Felt. Precision Processes (Textiles). *BP* 723,559–61

Improved results compared with those obtained by use of the process described in *BP* 614,271 (J.S.D.C., 65, 317 (June 1949)) are produced if an aqueous solution of a bromate is used at pH < 3 in presence of (*a*) a bromide or (*b*) formaldehyde or (*c*) formaldehyde sulphoxylate and/or thiourea and/or glyoxal and/or an alkali metal sulphide and/or an alkaline earth metal sulphide and/or a thiocyanate and/or a hydrosulphite. C. O. C.

Reducing the Felting Properties of Wool. Kroy Unshrinkable Wools. *USP* 2,671,006

The materials are treated with a hypochlorite solution containing 0·035–0·09% available Cl at pH 2·2–3·5 and 4–24°C. for ≮ 30 sec. preferably for 45 sec. C. O. C.

Treating Wool with Aqueous β-Propiolactone. U.S. Secretary of Agriculture. *USP* 2,672,397

The reaction between wool and β-propiolactone is greatly accelerated by presence of water the optimum effect being obtained in presence of ≮ 25% water (including the water present in the wool) on the dry weight of the wool. C. O. C.

Cloth Steaming. David Geasner Co. *USP* 2,670,622

Cloth, usually of wool, is wound under tension together with an apron of canvas on to a perforated drum. Steam is then admitted into the drum and a pressure of 2–3 lb. maintained at the inner surface of the wound cloth until the steam has penetrated through it. The rate of steam flow is then reduced, usually to about one-quarter, and the pressure at the inner surface may drop to 1·5–2·0 lb. This lessened flow and lower pressure is maintained for a short time, e.g. 2–4 min. The steam is then shut off and the drum connected to a large vacuum or air pump which exhausts the steam and any condensate from the cloth and draws air through it to cool it. After, say, 2–4 min. the partly dried and cooled cloth may be removed from the drum when it will be found that a superior surface finish has been imparted to it. C. O. C.

Reducing the Porosity of Fabrics. Wingfoot Corpn. *BP* 725,343

The fabric is "steam blasted", i.e. it is suddenly treated with heat at 138–275°F. higher than the b.p. of the liquid contained in it so that the liquid is instantaneously vaporised. At the same time that the heat is applied the fabric is under pressure so that the expansive force of the vapour is concentrated warp and weft ways. This causes the fibres to spread and close the weave. In an example a Fortisan fabric completely wet out with water was passed through calender rolls maintained at 350°C., as control a dry sample of the fabric was also passed through the rolls. The air porosity of the untreated fabric was 56·3 cu. ft./min./sq. ft., that of the sample calendered dry 13·6 and that of the steam blasted sample 1·3 (tested by procedure given in National Bureau of Standards Research Paper *RP* 1471, *J. Res. Nat. Bur. Standards*, 28, (May 1942)). C. O. C.

Organic Polysiloxane Resins for Rendering Textiles Water-repellent. Monsanto. *BP* 723,989

When using organopolysiloxane resins for rendering textiles water-repellent, presence as a catalyst of an alkyl titanate enables curing to be effected at < 150°C. C. O. C.

Flame-resisting Finish for Cellulosic Materials and increasing Adhesion to Rubber. BrC. *BP* 722,826

Impregnation with 2–3% of Ba or Ca oxalate gives a good flame-resisting finish without much loss in tenacity. The treated material shows greatly enhanced adhesion to rubber. C. O. C.

Preventing Deterioration in Cellulosic Wrappings around Nitrogenous Explosives. Celanese Corpn. of America. *BP* 725,800

Deterioration and loss of strength in cellulosic wrappers around nitrogenous explosives is greatly reduced by incorporation in the fibres of a water-insoluble, non-resinous organic compound which reacts with a nitrogen oxide to form a nitroso compound and/or a nitrile, e.g. diphenylethylene diamine. C. O. C.

Vapour Permeable, Liquid Impermeable Glass Fibre Fabrics. Owens-Corning Fiberglas Corpn. *USP* 2,673,823–5

Glass fibres are distributed as a matrix throughout a continuous phase of a high polymeric substance impervious to water and certain other liquids. Some or all of the fibres have been previously coated with a substance incompatible with the high polymer with the result that pores are formed at the interface between the two materials. The fibres may be either loose or a carded lap or yarn or fabric. After formation the high polymer is caused to flow to reduce the size of the pores. The fabric may be treated with either a hydrophobic agent to increase the water repellency or with a hydrophilic substance to increase the permeability. C. O. C.

Rendering Fabrics, Films, etc., Water-repellent, Greaseproof and Moistureproof. American Viscose Corpn. *USP* 2,672,427

The material is coated with an aqueous emulsion of a thermosetting resin, e.g. urea-formaldehyde, a water-soluble salt of a carboxyalkyl cellulose ether, and a wax. The coating is then treated with an aqueous solution of a metal salt with which the cellulose ether forms an insoluble salt. Finally the treated fabric is washed and dried at a temperature which sets the resin. C. O. C.

Flameproofing Cellulosic Materials. Titan Co. *BP* 723,451

Modification of *BP* 698,742 (J.S.D.C., 70, 34 (1954)), the process being now applied to materials other than cloth. C. O. C.

Coating Cloth and Paper with High Melting Point Thermoplastic Material. P. J. Massey. *BP* 724,274

Cloth or paper is coated with thermoplastic having viscosity at 190°C. at least equal to that of polyethylene of mol. wt. 19,000 and having a minimum working temperature > 240°F. by drying it to zero moisture content and then passing it through the nip formed by a rotating heated roll and a stationary heated roll. The thermoplastic is fed to the nip so as to form a roll-like mass which does not contact the cloth or paper until the nip is reached. C. O. C.

Pile Fabrics Backed with Foam Rubber. Dunlop. *BP* 724,078

Fibres are implanted on a foamed rubber latex backing by means of an electrostatic field. After vulcanising the backing is thicker, more durable, and also air permeable. J. W. B.

Coloured Glass Fibres. Owens-Corning Fiberglas Corpn. *USP* 2,671,033

Glass fibres are treated as strand or fabric with an organo metal compound which yields colourless or relatively white products when decomposed at high temperature on the glass fibres, e.g. stannic naphthenate. This renders the fibres highly receptive to pigments and dyes. C. O. C.

Thermoplastic Resinous Coatings having Coloured Patterns. Dunlop Rubber Co. *BP* 723,644

A mixture of a thermoplastic resin and a plasticiser in which it is insoluble at atmospheric temperature is applied as a pattern to a supporting surface. A granular mixture of a pigment, a thermoplastic resin and a plasticiser is then applied to the coating which is then heated to gel it and soften the granules, the hot material then being pressed. Subsequently the process is repeated using different colours on other parts of the support. C. O. C.

Luminescent Laminated Material. J. Friday. *BP* 723,789

A laminated material contains as one element radio-active ZnS evenly distributed in a set layer of amyl acetate lacquer, another element consists of a light-coloured backing to which the main lamination is adhered, while another consists of cellulose acetate lacquer borne on the exposed surface of the backing lamination. C. O. C.

Permanent Waving Composition (III p. 256).
Pastes for Coating, Printing, Finishing and Imparting Water-repellency (III p. 256).
Progress in Dye Manufacture and in Dyeing, Printing, and Finishing in the U.S.S.R. (IV p. 257).
Effect of Chlorine on Dyed Nylon (VIII p. 268).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Comparative Study of Cellulose and the Galactan of the White Lupin. L. M. Vinogradova, A. A. Konkin, and Z. A. Rogovin. *J. Appl. Chem. U.S.S.R.*, **27**, 1302–1306 (Dec. 1954).

The galactan of the white lupin—shown by Hirst, Jones, and Walder (see J.S.D.C., **63**, 249–254 (1947)) to be a 1:4-linked linear polymer of β-D-galactopyranose residues—is compared with cellulose of approx. the same mean D.P. (100 and 120 respectively). The galactan, which unlike cellulose is water-soluble, has a much higher heat of swelling in 96% alcohol, has a higher sorptive capacity for water vapour, and is much more rapidly acetylated than cellulose. The triacetate, unlike cellulose triacetate, is not completely sol. in chloroform, and the trinitrate is only slightly sol. in acetone; these facts are explained by the presence of a few carboxyl groups in the galactan mol., which react with hydroxyl groups of other galactan molecules.

Abstractor's Note—Although β-linked, this galactan, in having all the primary alcohol groups and glucosidic oxygens oriented on one side of the chain (and not alternately on one side and the other), may be considered to be more analogous to amylose than to cellulose.

A. E. S.

Fractional Dissolution of Nitrated Carbohydrates in Native Fibres. E. Dymling, H. W. Giertz, and B. G. Rânby. *Svensk Papperstidning*, **58**, 10–15 (15 Jan. 1955).

In attempting to separate wood carbohydrates (hemicellulose) from cellulose in delignified pulps using fractional dissolution, the fibres (sulphite and sulphate pulps, ramie and straw pulps) were nitrated in a mixture of nitric acid, phosphoric acid, and phosphorus pentoxide and then extracted with mixtures of ethyl acetate and ethyl alcohol of increasing ethyl acetate concn. A 35% ethyl acetate solvent yielded an easily soluble fraction and a fibrous residue. After denitration, and hydrolysis to monosaccharides, paper chromatography showed the first fraction to contain large amounts of glucose, mannose, and xylose. Fraction II was mainly glucose. The results obtained support the assumption that wood contains a well defined resistant framework of pure cellulose.

S. V. S.

Thermoplastic Water-soluble Synthetic Gum. G. K. Greminger, R. W. Swinehart, and A. T. Maasberg. *Ind. Eng. Chem.*, **47**, 156–160 (Jan. 1955).

A new cellulose derivative, hydroxypropylmethylcellulose containing 10–14% hydroxypropoxyl and 25–30% methoxyl substitution, is soluble in water and in certain (chiefly hot) organic solvents. The ether may be fabricated by thermoplastic techniques, e.g. injection and compression moulding, extrusion, hot casting from non-aqueous solvents, to provide sheets, filaments, mouldings, etc., which are soluble in water. W. K. R.

Decomposition of the Ethylenediamine–Cellulose Complex by Solvents. L. Loeb and L. Segal. *J. Polymer Sci.*, **15**, 343–354 (Feb. 1955).

Non-polar solvents which do not contain oxygen have little effect on the complex. Polar oxygen-containing solvents almost completely remove the diamine and cause a reconversion of the crystal structure to Cellulose I crystalline modifications. It is shown empirically that the directive influence of the solvent is related to its polarity and the temperature. W. R. M.

PATENTS

Precipitating Dispersed Solids. Industrial Tape Corpn. *BP* 723,868

A process for precipitating solids from a colloidal system comprises mixing the colloidal system from 1 part per million to 1% by weight of a water-soluble condensation polymer formed under non-acid conditions or a water-soluble polymer formed otherwise than by condensation, the polymer comprising groups capable of substantial ionisation and having ions carrying a charge opposed to the charge of the dispersed phase of the colloidal system. If cationic, the polymer may be a primary, secondary, or tertiary amine or a quaternary ammonium salt, whilst if anionic, the polymer may be a water-soluble polymer of an organic acid. S. V. S.

Paper Sizing. Industrial Tape Corpn. *BP* 723,869

A process for making sized fibrous webs from an aqueous dispersion of fibres and an anionic sizing agent comprises precipitating the sizing agent on the fibres by adding to the dispersion from 5 to 1000 parts per million of a dispersion of a water-soluble N-basic cationic organic polymer salt having a series of polar chemical bonds along the polymer chain capable of substantial ionization in water. The cationic polymer may be a primary secondary, or tertiary amine or a quaternary ammonium salt.

S. V. S.

Fibrous Webs. Industrial Tape Corpn. *BP* 723,901

A process for making fibrous webs from an aqueous dispersion of fibre and a colloidally dispersed resinous polymer or elastomer comprises mixing the fibre and polymer dispersions and adding one to 2,000 parts per million of a cationic water-soluble N-basic organic polymer salt comprising groups capable of substantial ionisation in water and furnishing ions having a charge opposed to the charge on the colloidally dispersed polymer, thereby causing precipitation. A web in which the precipitated solid is incorporated with the fibres is then formed.

S. V. S.

Stabilised Cellulose Ester Compositions. Eastman Kodak Co. *USP* 2,670,302

Cellulose esters are stable at ironing or moulding temperatures if any SO_4 radicals they contain have been neutralised and if a little of a glycidyl ether is present as a stabiliser, e.g. resorcinol diglycidyl ether. C. O. C.

Sizing of Paper (X p. 270).
Rendering Fabrics, Films, etc., Water-repellent, Grease-proof and Moistureproof (X p. 271).
Flameproofing Cellulosic Materials (X p. 271).

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Stains on Aniline-dyed Leather Uppers. Anon. *Leather World*, **46**, 517 (1954): *J. Amer. Leather Chem. Assocn.*, **50**, 53 (Jan. 1955).

Since aniline-dyed shoe uppers are not pigmented, they stain quite readily. One of the commonest causes of stains is dextrin from fabric linings. Another common cause is dye stain from the leather itself, while a third cause is tans from the insole or outsole. In 18 pairs of shoes examined recently by the Shoe and Allied Trades Research Association, over half were affected by dextrin. The lined shoes from four firms had faille-swansdown rubber combined linings, while some had swansdown plumpers in the ramp also. The fifth firm used a heavy drill with a swansdown plumper. There was always one layer of swansdown, and sometimes two layers beneath the vamp. Most swansdowns contain some organic dressing, usually starch and dextrin. The water extraction of the swansdown taken from the stained shoes showed 6–13% of these materials to be present; hence it was apparent that they were largely responsible for the stains. Toe puffs of the prepared type, celluloid-impregnated on one side, and containing 7–10% of organic dressing, showed evidence of staining. Laboratory tests indicated that if wetting took place from the edge or from the grain side of the leather, the toe puffs may stain more readily than a swansdown plumper with an equal amount of organic dressing. Evidence also indicated that water-soluble dyes, as well as oil-soluble ones, may migrate within the leathers and thereby cause stains. Recommendations are made to avoid wear complaints. These are—(1) make sure the leather contains no loose dye; (2) use linings and plumpers with the lowest possible content of organic dressing; (3) avoid toe puffs containing organic solubles. C. J. W. H.

Liberation of Aspartic Acid during the Acid Hydrolysis of Proteins. S. Blackburn and G. R. Lee. *Biochem. J.*, **58**, 227–231 (Oct. 1954).

Aspartic acid is preferentially liberated during hydrolysis of proteins with hot dilute acids but not with conc. HCl at 37°C. The specificity of the reaction and its mechanism are discussed. P. G. M.

PATENTS

Water-repellent Compositions for Impregnating Leathers. Dow Corning Corpn. *USP* 2,672,455

A mixture of (1) 15–50% by wt. of a compound of formula $Ti(OR^1)_4$ or an aliphatic hydrocarbon-soluble partial hydrolysate thereof (R^1 = aliphatic hydrocarbon of < 13 C which contains 1–3 OH groups), (2) 5–7% by wt. of a methylpolysiloxane resin composed of trimethylsiloxane and SiO_2 units so that CH_3:Si::1·0–2·5:1·0, and (3) 15–80% by wt. of polysiloxanes of formula $R^2_{2n}{}^2Si_{2-n}$ (R^2 = Alk or alkenyl of < 4 C or a monocyclic Ar; n = on average 1·0–1·45) can be applied to any kind of finished or unfinished leather to impart a resistance to water penetration much greater than any hitherto possible. C. O. C.

Treating Hides and Skins with a Vinyl Ether–Maleic Anhydride Copolymer. G. *BP* 725,671

The hides, etc., are treated in presence of water with a vinylalkyl or subst. alkyl ether–maleic anydride copolymer in which 5–15% of the anhydride groups have been converted to the ammonium salt—half amide. The treatment is applied before, during or after chrome tanning. It gives body and fullness to the leather and causes no alteration in affinity for dyes, full even dyeings, including blacks, being easily obtained. With suède leather they enable a fine uniform nap to be produced. *BP* 725,672

Similar effects are obtained if the polymer has not had its anhydride groups converted. C. O. C.

Rapid Testing and Numerical Evaluation of the Covering Power of Casein and Collodion Opaque Colours (XIV p. 275).

Determination of Amide Nitrogen in Collagen and other Proteins (XIV p. 276).

XIII—RUBBER; RESINS; PLASTICS

Some Problems involved in the Colouring of Plastics. C. H. Hall. *Plastics Inst. Trans.*, **23**, 4–15 (Jan. 1955).

A discussion of the problems involved in colouring plastics with organic pigments which are soluble to some extent in the polymer, the plasticiser or some other component of the plastics material, special attention being paid to colour-bleeding, blooming and chalking. C. O. C.

Double Stabiliser Combinations for Chlorine-containing Polymers. W. G. Vennels. *Plastics Inst. Trans.*, **23**, 44–60 (Jan. 1955).

A description of work done on heat stabilising combinations for polyvinyl chloride based on metal soaps. Data are given which illustrate the results of the synergistic activity of various metal soap combinations and of some metal soaps used together with epoxy resin, basic lead carbonate, inorganic stabilisers and certain zinc soaps. They are compared with the results obtained with some proprietary materials as well as some organo-tin compounds and known inorganic stabilisers. C. O. C.

Weathering of Polyvinyl Chloride. J. B. DeCoste and T. V. Wallder. *Ind. Eng. Chem.*, **47**, 314–322 (Feb. 1955).

Unprotected polyvinyl chloride is degraded rapidly on atmospheric exposure, but if the formulation is such that the body of the plastic is protected from sunlight, compositions resistant to lengthy exposure can be achieved. The stabiliser, plasticiser, and pigment in a polyvinyl chloride system have each an important influence on weather-resistance. Stabilisers, e.g. dibasic lead phosphite, and pigments, e.g. channel black and titanium dioxide, which have high opacity to light transmission, provide the best protection. Monomeric plasticisers of low volatility have satisfactory outdoor permanence provided that they are adequately shielded by a suitable pigment. Resistance to photo-oxidation is also required in a satisfactory plasticiser. W. K. R.

PATENTS

Cork Composition Surface Covering. Armstrong Cork Co. *USP* 2,671,733

Granules of cork 80–90% of which are > 10 mesh, the remainder being < 20 mesh are coated with a colouring material and then bonded in close face-to-face contact by about 1·2–4·8% of a binder based on the compressed volume of the ink. This gives a unique coloured surface which can be obtained in no other way. C. O. C.

Embossed Thermoplastic Film. M. A. Chavannes. *BP* 725,436

Deep and uniformly embossed patterns are rapidly produced on thin film by using a perforated or porous embossing surface which either goes around or forms part of the surface of a roller. The interior of the roller is divided into two sections, one where suction is applied and the other where there is no suction. The hot film is drawn on to the embossing surface by the suction and then passes on to the second section of the roller where it is cooled and stripped off. C. O. C.

Foils and Coatings from Polyvinyl Chloride Pastes (III p. 257).

Chlorine-containing Vinyl Polymeric Compositions (VI p. 266).

Polyacrylonitrile–Polyaminoethylvinyl Ether Mixtures having Affinity for Acid Dyes (VI p. 266).

Urea–Formaldehyde Condensation. IX—The Presence of Dimethylene Ether Bridges in Condensates (X p. 270).

Stabilised Cellulose Ester Compositions (XI p. 272).

XIV—ANALYSIS; TESTING; APPARATUS

Direct Ethylenediamine Tetra-acetate Titration Methods for Magnesium and Calcium. C. W. Gehrke, H. E. Affsprung, and Y. C. Lee. *Anal. Chem.*, **26**, 1944–1948 (Dec. 1954).

A direct method for Mg is described, in which ethylenediamine tetra-acetate is the titrant, and Eriochrome Black T the indicator. The Ca is pptd. as $CaSO_4$ at pH 5–7, and is removed by filtration. The same titrant is used, with murexide as indicator, for the direct titration of Ca; interfering anions are removed on a suitable anion-exchange column. The method is equal in accuracy to the classical oxalate–permanganate method, and is more rapid. The procedure is given in full. J. W. D.

Precise Measurements of Particle Surface Area with the Microscope. F. D. Pidgeon and C. G. Dodd. *Anal. Chem.*, **26**, 1823–1828 (Nov. 1954).

A graticule projected-area method is described, which is direct and accurate; the need for measuring or assuming "shape factors" and for determining suitable statistical methods for averaging size-distribution data (as in measurements of particle "diameter") is eliminated. Mounting techniques have been developed which permit extension of the method to particles of sub-sieve size. Comparisons are reported which show that results obtained with crushed quartz powders, agree well with those obtained by gas adsorption and liquid permeation, providing that there are few particles < 1–3μ. An extension of the method to particles < 1μ. is suggested. J. W. D.

Analysis of Aqueous Mixtures of Hydrogen Peroxide and Aldehydes. C. N. Satterfield, R. E. Wilson, R. M. LeClair, and R. C. Reid. *Anal. Chem.*, **26**, 1792–1797 (Nov. 1954).

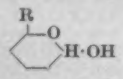
The preferred methods are— liberation of I_2 with glacial acetic acid–HI for total peroxide, oxidation by alkali–H_2O_2 for total aldehyde, and colorimetric determination with Schiff's reagent after reduction of the peroxides, for H·CHO. Full details are given. J. W. D.

Photometric Titration of Weak Acids. R. F. Goddu and D. N. Hume. *Anal. Chem.*, **26**, 1679–1684 (Nov. 1954).

Photometric titration for the determination of weak acids (or bases) which differ in light-absorption characteristics in the ionised and unionised forms has been studied by theory and experiment. Satisfactory end-points are obtained only if the product of ionisation constant and concn. $\geq 10^{-12}$ at concn. $\geq 10^{-2}$M. The titration of various substituted phenols is used to demonstrate the capabilities and limitations of the method in the determination of weak acids individually, in mixtures, and in the presence of strong acids. The photometric method has advantages over potentiometric methods when determinations are made in highly dil. soln., and with very weak acids. J. W. D.

Approach to the Correlation of R_f Value with Structure in the Paper Chromatography of Carbohydrate Compounds. M, F. Levy. *Anal. Chem.*, **26**, 1849–1850 (Nov. 1954).

The work of Isherwood and Jermyn (*Biochem. J.*, **48**, 515 (1951)) has been examined. It is assumed that aldoses, aldomethyloses, and aldohexuronic acids have, in common, a pyranose structure—



A function $f(R_f)$ has been sought such that—

$$f[R_f(b)] - f[R_f(a)] = K$$

for homomorphous pairs, K being constant for all such pairs, and dependent upon the change in R group in passing from one member of the pair to the other. Some success is reported with the transition: aldose → aldomethylose, but with a number of others K has not been found to be constant. The study is reported in some detail. J. W. D.

Dye Displacement Technique for Velocity Distribution Measurements. J. K. Ferrell, F. M. Richardson, and K. O. Beatty. *Ind. Eng. Chem.*, **47**, 29–33 (Jan. 1955).

By means of a new dye displacement technique, the velocity distribution of a fluid in laminar flow in a smooth circular tube has been measured to within 0·002 in. of the wall of a 0·5-in. tube. The results agree with the parabolic velocity distribution predicted by the theory of laminar flow. Measurements even closer to the tube wall would be possible if molecular diffusion of the dye could be reduced. W. K. R.

Standard Tests for Lubricating Oils. Dr. Schiffner. *Textil-Praxis*, **10**, 165 (Feb. 1955).

Proposed standard tests for lubricating oils should include storage properties, solubility, emulsifying properties, pH, cation activity, ability to wash out, active content, Mackey test, and flash point. B. K.

Colour Reactions of Carbazole, Indole, Pyrrole, and their Derivatives with 2-Bromo-2-nitroindan-1:3-dione. G. Vanag. *J. Anal. Chem. U.S.S.R.*, **9**, 217–219 (July–Aug. 1954)

2-Bromo-2-nitroindan-1:3-dione gives characteristic colours in glacial acetic acid with many cpd. containing a pyrrole ring. Of the reactions examined, that for carbazole is the most sensitive (red colour, detectable at 2 p.p.m.), and that for tryptophan the most specific (pink with greenish fluorescence, detectable at 10 p.p.m.). A. E. S.

Chloramine B as a Volumetric Reagent—Quantitative Bromination of Phenols and Aromatic Amines. A. Singh. *J. Indian Chem. Soc.*, **31**, 605–608 (Aug. 1954).

The method of Koppeschaar (*Z. anal. Chem.*, **15**, 2531 (1876)) as modified by Day and Taggarat (*Ind. Eng. Chem.*, **20**, 545 (1928)) has been successfully applied to the bromination of phenols and aromatic amines with Chloramine B in presence of excess KBr in acid soln. Three procedures are given in detail, and the best of these is defined by numerous experimental results, which are tabulated. J. W. D.

Potentiometric Determination of Aromatic Amines and Phenols using Chloramine B as a Brominating Agent. A. Singh. *J. Indian Chem. Soc.*, **31**, 609–611 (Aug. 1954).

Aromatic amines and phenols may be directly titrated with a standard soln. of Chloramine B in presence of excess KBr and HCl; the end-point may be clearly detected potentiometrically. Full experimental details are given, and numerous proving analyses are quoted. J. W. D.

Chromatography of Dye Intermediates. II—Paper Chromatography of Naphtholsulphonic Acids. J. Latinák. *Chem. Listy*, **48**, 1354–1359 (1954): *Chem. Abs.*, **49**, 619 (10 Jan. 1955).

Naphtholsulphonic acids and sultones of 1-naphthol-8-sulphonic acids were successfully separated by paper chromatography. The R_f values are influenced mainly by the number of SO_3H groups in the molecule and by the relative positions of the SO_3H and OH groups. Chromatography was done on Whatman No. 4 paper by the descending method at 19°c, using as solvents butyl alcohol–acetic acid–water 4 : 1 : 5 (system I) and butyl alcohol–pyridine–water (system II). Detection was done by coupling with diazonium salts or by fluorescence under ultraviolet radiation. Sultones were hydrolysed with 5% KOH prior to the detection. Compounds, positions of OH and SO_3H groups, R_f in systems I and II, and fluorescence are: 1-naphthol-3:6:8-trisulphonic acid, 1–3:6:8, 0·02, 0·02 white–blue; α acid, 1–3:8, 0·10, 0·14, white–blue; δ acid, 1–4:8, 0·09, 0·21, white–blue; 1-naphthol-2-sulphonic acid, 1–2, 0·79, 0·80, violet; 1-naphthol-3-sulphonic acid, 1–3, 0·61, 0·69, dark blue; Nevile & Winter acid, 1–4, 0·52, 0·67, dark violet; naphthol-L acid, 1–5, 0·50, 0·67, white–blue; Schölkopf acid, 1–8, 0·72, 0·77, blue–white; 1-naphthol, 1–0 0·95, 0·93, —; 2-naphthol-3:6:8-trisulphonic acid, 2–3:6:8, 0·02, 0·02, light green; R acid, 2–3:6, 0·04, 0·11, light blue; G acid, 2–6:8, 0·05, 0·12, light blue; oxy-Tobias acid, 2–1, 0·71, 0·80, —; Schäffer acid, 2–6, 0·47, 0·66, dark blue; F acid, 2–7, 0·48, 0·66, blue–violet; Crocein acid, 2–8, 0·54, 0·70, bright light blue; 2-naphthol, 2–0, 0·94, 0·93, dark violet; and sultones of 1-naphthol-3:6:8-trisulphonic acid, 0·17, 0·23, yellow; 1-naphthol-3:8-disulphonic acid, 0·71, 0·73, green–yellow; and 1-naphthol-8-sulphonic acid, 0·96, 0·96, yellow. C. O. C.

Coal-tar Colours. XVIII—FD&C Yellow No. 1. C. Stein and K. A. Freeman. *J. Assocn. Off. Agric. Chem.*, **37**, 523–527 (1954): *Chem. Abs.*, **49**, 102 (10 Jan. 1955).

Titration with $TiCl_3$ and spectrophotometric analysis in 0·1 N-NaOH have been proved to be satisfactory methods for estimating the dye. Complete analyses of commercial samples show that the specifications in the U.S. Coal-tar Colour Regulations are accurate and practical. C. O. C.

Aromatic Diazo Compounds. XII—Identification of Primary Aromatic Amines by means of the Absorption Spectra of the Corresponding Azo Dyes. Z. J. Allan and F. Mužík. *Chem. Listy*, **47**, 380–391 (1953): *Chem. Abs.*, **49**, 207 (10 Jan. 1955).

Rapid identification of aromatic primary amines is based on diazotising them, coupling with R salt (or resorcinol), and measuring the absorption spectra of the resultant azo dyes. Conditions of diazotisation and coupling are laid down in detail and absorption spectra of nearly 200 azo dyes listed. Effect of constitution, especially of the coplanarity of azo groups, on the absorption spectra is indicated. C. O. C.

Spectrophotometric Determination of Benzanthrone and 2:2′-Dibenzanthronyl. K. B. Whetsel. *Anal. Chem.*, **26**, 1974–1977 (Dec. 1954).

The absorbances of soln. of benzanthrone (I) and 2-2′-dibenzanthronyl (II) in conc. H_2SO_4 are measured at 374 and 550 mμ., and the following equations are applied—

$$\% \text{ Benzanthrone} = 119{\cdot}8\,A_{374\text{m}\mu} - 134{\cdot}0\,A_{550\text{m}\mu}$$

$$\% \text{ Dibenzanthronyl} = 231{\cdot}9\,A_{550\text{m}\mu} - 24{\cdot}5\,A_{374\text{m}\mu}$$

where $A = \log (I/T)$. Dibenzanthrone interferes seriously at both wavelengths, but may be detected and determined from its absorbance at 850 mμ., at which wavelength neither I nor II shows measurable absorption. Traces of HNO_2 greater than those permitted in analytical-grade H_2SO_4 markedly affect the spectrum of II. The method is applicable over a wide range of concn. J. W. D.

Separation of Amaranth and Tartrazine. G. G. McKeown. *J. Assocn. Off. Agric. Chem.*, **37**, 527–529 (1954): *Chem. Abs.*, **49**, 619 (10 Jan. 1955).

Adsorb the dyes from solution in dil. HCl. on a column of alumina. Elute the Tartrazine with dil. Na acetate and the Amaranth with dil. NaOH. Determine the recovered dyes spectrophotometrically. C. O. C.

Determination of Fabric Weight. DIN 53,854: *Melliand Textilber.*, **35**, 1395 (Dec. 1954).

German standard method. S. R. C.

Swelling Techniques in the Photomicrography of Textile Fibres. W. Bobeth. *Z. ges. Textilind.*, **57**, 15–22 (5 Jan. 1955).

The use of various inorganic and organic compounds in the detection of synthetic fibres by photomicrography is described with special reference to substances which cause the fibres to swell in a characteristic manner. B. K.

Preparation of Cross-sections and Imprints of Fibres. W. Seffens. *Textil-Praxis*, **10**, 3–8 (Jan. 1955).

Details are given of a modified method of preparing cross-sections and imprints of fibres using cellulose nitrate and similar films. 33 illustrations of various fibres are given. B. K.

Microreactions of Fibres with Sulphuric Acid. W. Bobeth. *Textil-Praxis*, **10**, 165–166 (Feb. 1955).

The behaviour of a wide range of natural and synthetic fibres in sulphuric acid is described with respect to its application for identification of the fibres under the microscope. B. K.

Determination of the Molecular Weight of Polyacrylonitrile. H. Frind. *Faserforsch. und Textiltech.*, **5**, 540–553 (Dec. 1954).

From viscosity and osmotic pressure measurements on solutions of various polyacrylonitrile preparations in dimethylformamide, the wt.-average mol. wt. $\bar{M}_w$ (calc. by means of Houtz's formula $[\eta] = 1{\cdot}75 \times 10^{-3} \bar{M}_w^{0.66}$ (*Text. Research J.*, **20**, 786 (1950)) and number-average mol. wt. $\bar{M}_n$, respectively, are determined. The polyacrylonitrile preparations vary greatly in $\bar{M}_n$ (9,000–69,000), but the inhomogeneity $(\bar{M}_w/\bar{M}_n - 1)$ is substantially const. at 0·7. It is shown that, if the wt.-average mol. wt. is used, the frictional-const. method of determining molecular dimensions can be applied to unfractionated polymer to give the dimensions in a homogeneous fraction of mol. wt. $\bar{M}_w$. The degree of coiling of the polymer (ratio of stretched-out length to the root mean square of the actual distances between the ends of molecules) increases with mol. wt. (from 3·8 to 10·6). A. E. S.

Detergency Measurements using Artificially Soiled Cloths. F. L. Diehl and J. B. Crowe. *J. Amer. Oil Chem. Soc.*, **31**, 404–408 (Oct. 1954).

Four types of artificially soiled cloths were compared for their utility in the evaluation of the cleaning power of representative detergent products. Sensitivity and reproducibility of the various soiled cloths in measurement of soil removal and whiteness retention were studied. The results were compared by means of naturally soiled cloths. P. G. M.

Flame Photometry Control Methods for Bleaching Apparatus. E. Venemark. *Svensk Papperstidning*, **58**, 78–85 (15 Feb. 1955).

Uniform treatment of the pulp is very important in a continuous bleaching process and may be assisted by flame-photometric methods of determining alkali and Ca even in microquantities. The distribution of a solution of one of these elements in pulp stock, whether as bleach or as a tracer, may be studied in microsamples containing about 0·02 mg. of the metal. The weighed sample is extracted with 10 ml. of H_2O and 0·1 N-HCl, and the element determined in the liquor by flame photometry. The method can also be used with a tracer to study the passage of stock through a bleaching tower, the rate of bleaching, and the washing operation. S. V. S.

Seventy Years of Colorimetry. G. J. Chamberlin. *Amer. Dyestuff Rep.*, **44**, P 18–P 21, P 28 (3 Jan. 1955).

A brief historical survey of the Lovibond system of colour measurement is followed by a description of a method of relating it with the C.I.E. system of colour transformation. A modified Lovibond Tintometer can be employed to interconvert instantly between Lovibond glasses and Judd's uniform-chromaticity scale. J. W. B.

Measurements of Ultraviolet Dichroism. J. C. Ward. *Proc. Royal Soc.*, **228**, 205–219 (22 Feb. 1955).

The dichroism of ultraviolet absorption bands of thin single crystals of some organic compounds has been measured, and the results have been used to determine the direction of the molecular transition moments of the peptide group and of substituted phenyl groups. Some of the theory of the optics of absorbing crystals is presented in a form useful for interpreting measurements of dichroism in terms of crystal and molecular structure. W. R. M.

Hydrogen-bond Formation. II—Use of Refractive-index Measurements to detect Intermolecular Complex Formation in Solution. F. M. Arshid, C. H. Giles, E. C. McLure, A. Ogilvie, and T. J. Rose. *J.C.S.*, 67–79 (Jan. 1955).

The square of the refractive index may be used equally as well as the dielectric constant (cf. Part I, *J.C.S.*, 3799 (1952)) for detecting the ratio in which organic molecules associate intermolecularly in binary soln., with the added advantage that aq. as well as non-aq. soln. can be studied. In particular, hydrogen-bond complexes can be detected by this means, and typical results are given. Anion–cation association also is detectable, but not other forms of association. The method appears equally useful for detecting strong and weak complexes, is rapid and simple, requires only small amounts of substance (e.g. soln. as dilute as 0·01 M.), and appears to have some advantages over hitherto used procedures. Intramolecular as well as intermolecular bonds can be detected. H. H. H.

Assessment of Fastness Properties of Textiles. P. Rabe. *Textil Praxis*, **9**, 1165–1168 (Dec. 1954).

Various points are amplified in connection with the standard methods of fastness testing issued by the D.E.K. relating to the following tests—organic solvents, seawater, mercerising, hydrogen peroxide, potting, rubbing, acids, sulphur stoving, perspiration, soda boiling, cross-dyeing, alkaline milling, acid milling, and washing. Photographs of special apparatus recommended for the tests are included. B. K.

Acceptability and Perceptibility of Fading. AATCC, Northern New England Section. *Amer. Dyestuff Rep.*, **43**, P 885–P 890 (20 Dec. 1954).

Eighteen shades, strategically scattered throughout textile colour space, and with 3 degrees of fading, are assessed by eighteen observers, the average equivalence of fade from one region of colour space to another being determined in terms of the Adams–Nickerson colour difference formula. The problem of metamerism is studied in an experiment involving 14 samples and 8 observers. Results are tabulated; it is concluded that problems of metamerism must be taken into account when dyeing with formulae different from those submitted, that it is of doubtful value to attempt to refine instrumental methods to a greater degree than the precision implied by the variation involved, and that it would be unwise to employ the results of only one sight-test evaluation in cases of economic importance. J. W. B.

Rapid Testing and Numerical Evaluation of the Covering Power of Casein and Collodion Opaque Colours. O. Zohlen. *Leder*, **5**, 187 (1954): *J. Amer. Leather Chem. Assocn.*, **50**, 61 (Jan. 1955).

The following method was developed to measure the covering power of opaque colours. Make a stripe 0·5 cm. wide with India ink on a sheet (6 × 10 cm.) of white carton paper. On such a sheet, resting on a white paper base, put a round, glass (crystallising) dish with a flat bottom. The dish must have an internal diameter of 8 cm. and a surface area of the base of exactly 50·25 sq.cm. Add to the dish 50 c.c. of distilled water for testing casein colours or 50 c.c. of organic solvent, e.g. 50 parts of denatured alcohol and 25 parts each of butyl acetate and ethyl acetate, for collodion colours. The liquid must be exactly 1 cm. deep in the dish. Stir 1 g. of colour if of strong, or 2 g. if of weak, covering power, into water or solvent and make up to 100 c.c. Colours with strong covering power are black (carbon), brown (iron oxide), yellow or orange (lead chromate), and white (titanium dioxide); those with rather weak covering power are yellow, orange, or red (cadmium sulphide or selenide); scarlet, red and blue organic pigments have weak covering power. Add the freshly prepared 1% test solution from a graduated pipette to the dish with water or solvent until, after stirring with a small glass rod or the pipette tip,

the black stripe is not visible. Near the end-point additions should be made dropwise. Observations, using diffuse daylight, may be made by either of two methods: I—Put the dish directly on the paper with the stripe so that all light must pass through the solution. II—Place an inverted crystallising dish between the paper with stripe and the dish to which the test solution is being added; light then falls directly on the stripe. Both methods give reproducible results, but one may be better than the other depending on conditions and the colour being tested. Covering power D is inversely proportional to the volume required, therefore $D = 1000/x$, where x is the calculated volume of 1% test solution that would be required to hide the stripe if the dish had an area of 100 sq.cm. For example, 9·04 c.c. of blue dye was required to hide the stripe; $\therefore x = 9{\cdot}04 \times 100/50{\cdot}25 = 18$. $D = 1000/18 = 55{\cdot}5$. Results are given for a number of colours, some of which are shown in the table.

Colour	*Pigment Content*	D_I	D_{II}
Brown	30% Ferric oxide	102	84
Yellow	30% Chrome yellow	69	108
Yellow	13% Cadmium yellow	42	58
White	40% Titanium dioxide	167	245
Orange	13% Molybdenum orange	58	71
Scarlet	7·6% Organic pigment	43	59
Red	5% Organic Bordeaux	39	34
Black	6% Carbon Black	115	66

Method I gave values 40–45% higher than Method II for blacks, but Method II gave higher values than Method I by 28–32% for whites, by 30–38% for yellows, and by 20% for orange inorganic pigment products.

C. J. W. H.

Fractional Precipitation as a Method for Estimating the Chain-length Distribution of Celluloses of High Molecular Weight. T. E. Timell. *Svensk Papperstidning*, **58**, 1–9 (15 Jan. 1955).

Fractionations were carried out with a nitrated, raw ramie cellulose, with acetone $-H_2O$ as the solvent–non-solvent system. The degree of polymerisation of the fractions was calculated from the intrinsic viscosity value as determined at a rate of shear of 500 sec.$^{-1}$, and was in most cases adjusted to a standard N content of 13·6%. The total numbers of fractions isolated were respectively 12, 12, 168 and 35. Owing to degradation of the cellulose nitrate in acetone solution, the time of contact between polymer and solvent was kept to a minimum. The necessity for repeated refractionation to achieve good separation, and the instability of cellulose nitrates in solution, present great difficulties in the investigation of high-mol.wt. celluloses by this method. S. V. S.

Determination of Amide Nitrogen in Collagen and other Proteins. E. F. Mellon, S. J. Viola, and S. R. Hoover. *J. Amer. Leather Chem. Assocn.*, **49**, 710–719 (Nov. 1954).

A modification of the alkaline hydrolysis method of Warner and Cannan (*J. Biol. Chem.*, **142**, 725 (1942)) is described fully, and there is a diagram of the apparatus (which constitutes the main modification). Results obtained with a number of proteins are cited, and these show excellent agreement with the values quoted in other literature. Studies are reported which indicate that the amide nitrogen content of collagen derived from cowhide is not affected by the level of feeding, but that changes are brought about in both collagen and gelatin by subsequent processing. J. W. D.

PATENTS

Continuously Testing the Concentration of a Constituent present in a Liquid. British Electricity Authority. *BP* 721,884

Apparatus for monitoring the hardness of water leaving a water softening plant. C. O. C.

Colour Mixture Computer. General Aniline. *USP* 2,671,609

A computer for predicting the colour or tristimulus values of colour mixtures. C. O. C.

Testing the Resistance of Cloth to Abrasion, Flexing and Creasing. Asbestos Textile Institute. *USP* 2,670,627

Apparatus for testing the wearability of asbestos and other heavier cloths. C. O. C.

Transparency of Fabrics made from Different Types of Fibre (VI p. 264).

Factors which influence Fastness to Crocking [Rubbing] (VIII p. 267).

XV—MISCELLANEOUS

Textile Trade Publications. M. Matthes. *Melliand Textilber.*, **35**, 1375–1380 (Dec. 1954).

A number of textile reference works in German are listed. S. R. C.

Occupational Dermatitis in Government Workers. S. W. Becker. *J. Invest. Dermatol.*, **22**, 249–252 (1954): *Chem. Abs.*, **49**, 540 (10 Jan. 1955).

The most common causes of allergic contact dermatitis are the chemicals used in printing and in allied fields. Primary irritants such as organic solvents and cleansing agents are responsible for 43% of the dermatoses. A large number of patients acquire dermatitis from excessive use of soap. C. O. C.

PATENTS

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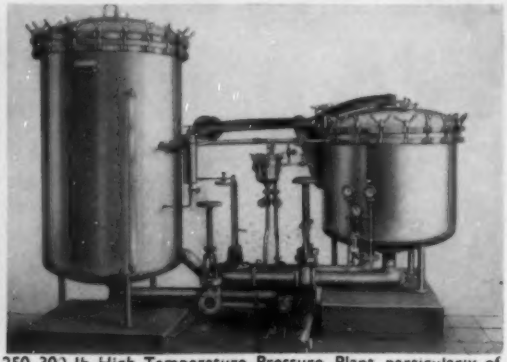

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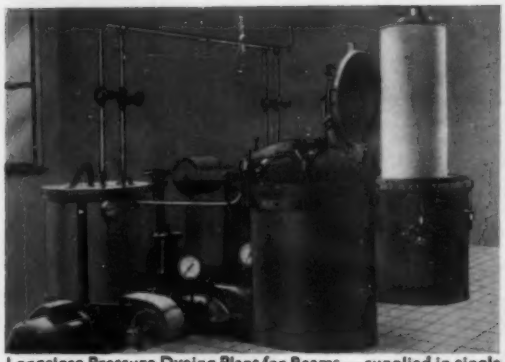

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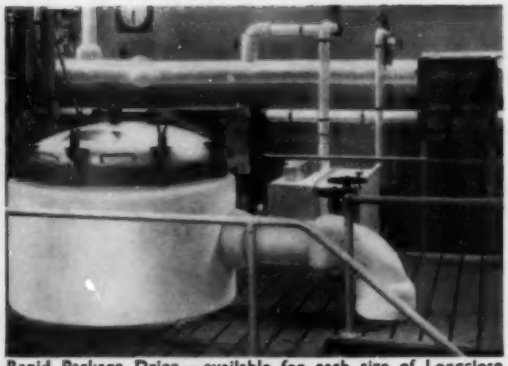

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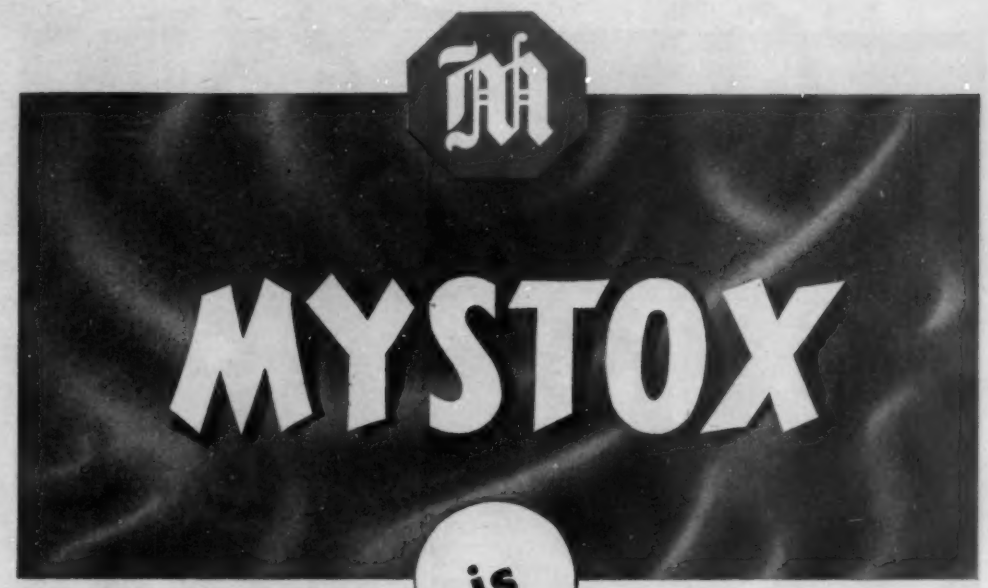

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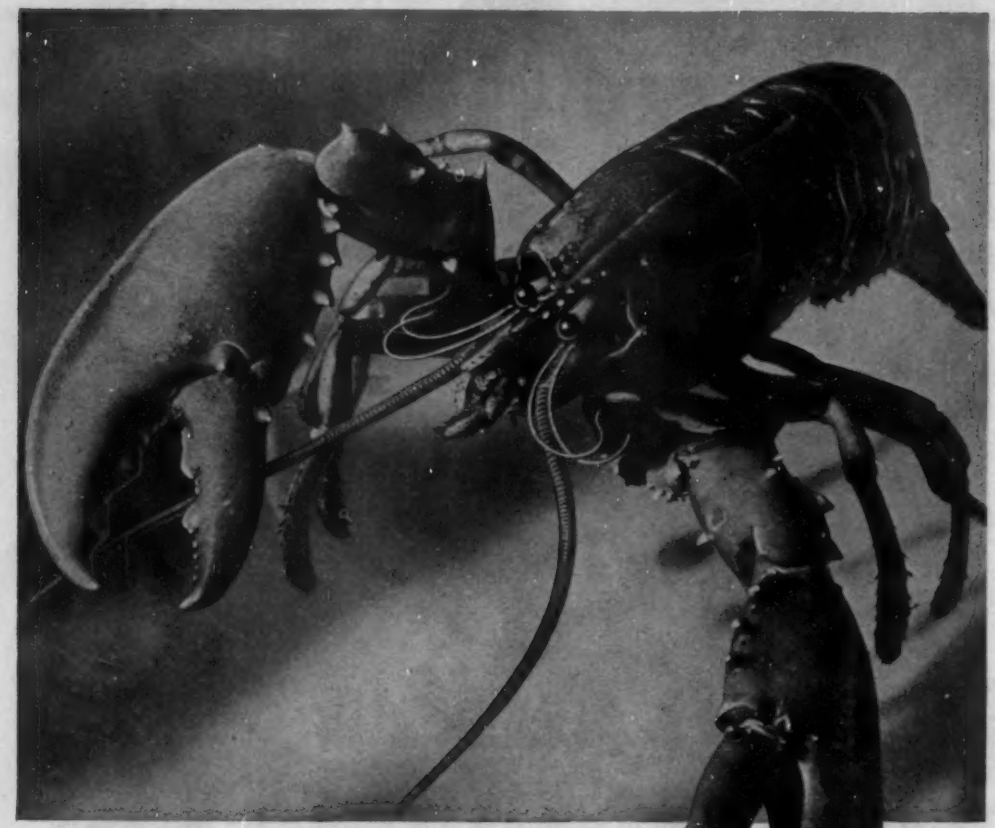

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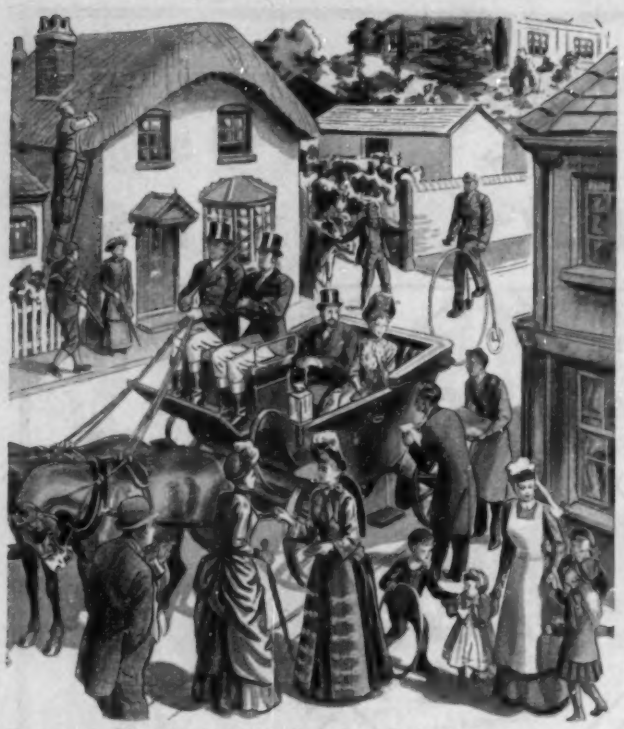

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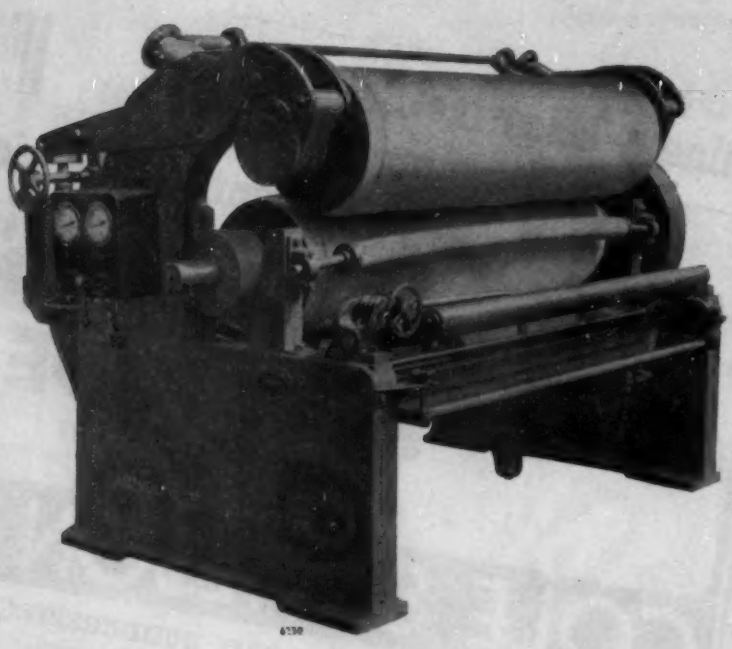

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